DEPARTMENT OF COMMERCE

TECHNOLOGIC PAPERS

OF THE

BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 116

SILICA REFRACTORIES

FACTORS AFFECTING THEIR QUALITY AND METHODS OF TESTING THE RAW MATERIALS AND FINISHED WARE

BY

DONALD W. ROSS, Assistant Ceramic Chemist
Bureau of Standards

ISSUED APRIL 19, 1919

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By Donald W. Ross

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I. INTRODUCTION

The tremendous trade in silica refractories for by-product coke ovens, steel and copper reverberatory furnaces, etc., which has sprung up in the last two years has caused attention to be turned to the need of complete information concerning the manufacture and properties of these materials and of specifications for their inspection.

Although considerable valuable research work has already been done in connection with silica refractories, it was thought desirable to review the information at hand and to do experimental work upon points which had not previously been as thoroughly worked out as would seem necessary.

II. REVIEW OF PREVIOUS WORK

In the study of previous work on silica refractories we find that the work of the Geophysical Laboratory of the Carnegie Institution[1] in connection with the physico-crystalline relations of the forms of silica and the silicates of the ternary system CaO–Al_2O_3–SiO_2 serves at once as a review and summary of most work on these subjects to date. Likewise, the work of McDowell[2] on the silica refractories may be considered as a practical summation of the previously published data on manufactured silica refractories. In this work is also presented a bibliography of the literature covering both the silica minerals and the manufactured ware.

[1] Stability Relations of the Silica Minerals, C. N. Fenner, Am. J. S., 36; October, 1913. The Ternary system CaO–Al_2O_3–SiO_2, Am. J. S., 39; January, 1915.

[2] A Study of the Silica Refractories, J. Spotts McDowell, Am. Inst. Mining Engineers, Bull. 119.

Hence, the individual references are not taken up here, except some that refer to varieties of raw silica brick materials and other data which have not received particular emphasis by previous writers. Such points of the above and later work are here considered as appear to have special bearing upon the present work.

In some cases the silica rock as mined is either hewn into blocks or crushed and used directly in furnace construction. Richter [3] states that Crummendorf schists which are very fine grained and exceptionally uniform in structure and impurities and contain from 95 to 96 per cent of silica are furnished in rough or sawn blocks. These are used directly as refractories in place of clay fire brick and are supposed to last longer. Some varieties contract in use; others expand slightly.

In the choice of raw materials for the manufacture of silica refractories, Khoetsky [4] has considered the subject from the standpoint of chemical purity, size of grain obtainable, and the magnitude of the increase in volume on heating. He concludes that to meet the temperature requirements in use the brick must not contain less than 96 per cent silica (SiO_2) (98 per cent silica for the raw material); that the rock must not yield too fine a powder on crushing; and, third, the order of merit of the natural forms of silica is given as chalcedony, old quartzites, and vein quartz. Quartz schists, sandstones, and sand are considered unsuitable, the first on account of their structure and the presence of many impurities in the form of inclusions, the two latter on account of their variability in composition and their excessive fineness after grinding.

The same problem has been attacked by determining the specific gravity [5] of the material before and after repeated heatings to cone 15–16 (1450° C approximately). The author designates (*a*) as good material, (*b*) as fair, and (*c*) as poor, and presents the following figures in Table 1:

TABLE 1.—Differences in Rate of Drop in Specific Gravity of Different Varieties of Silica Raw Materials on Being Heated Repeatedly to Cones 15–16

Heatings to cone 15–16	Specific gravity		
	(a)	(b)	(c)
Before heating	2.65	2.65	2.64
After first heating	2.40	2.47	2.53
After third heating	2.38	2.37	2.51
After fifth heating	2.38	2.37	2.43

NOTE.—(*a*) Transformed most readily, (*b*) required a somewhat greater heat treatment, and (*c*) had apparently not reached minimum attainable specific gravity by the end of the test.

[3] Hugo Richter, Zs. Ver. Zucherind, **65**, pp. 263–273; 1915.
[4] Rev. Soc. Russe de Metall, **1**, pp. 170–202; 1914.
[5] Anon, Tonind. Ztg., 38, pp. 163–165.

As seen from the figures, (*a*) practically completes its drop in specific gravity in one burn while (*c*) continues to drop for a long series of ignitions.

Concerning the rates of transformation of the different forms of silica to cristobalite on being heated, Endell and Rieke [6] state that when heated to 1400° C rock crystal was slowest in being transformed, while strongly twinned pseudomorphic quartz from the Taunus and pegmatitic quartz from Norway were found to be largely converted in two or three such heatings. Powdered silica glass, amorphous silica, and chalcedony were completely changed

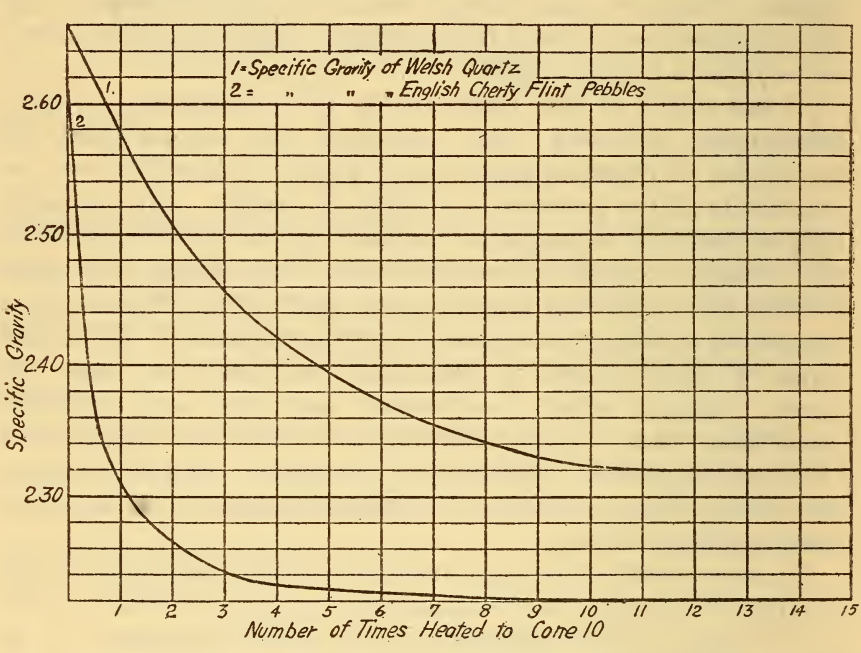

FIG. 1.—*Rate of drop in specific gravity of English flint pebbles and Welsh quartz, when repeatedly heated to cone 10 (after Mellor)*

after a single burning. The rate of transformation of silica glass into cristobalite was found to increase rapidly with the temperature from 1300° C upward.

In Fig. 1 are shown the results of work by Mellor [7] on the changes in specific gravity of quartz and English flint after repeated heatings to cone 10. The quartz was presumably Welsh and the flint was derived from flint pebbles of the chalky limestones of southeastern England or northern France. These flint pebbles are cryptocrystalline silica and their origin is practically identical with that

[6] K. Endell and R. Rieke, Zs. Angew, Chem., 25, pp. 2019–2020; 1912.
[7] J. W. Mellor and A. J. Campbell, Trans. Eng. Ceramic Soc., 15, pp. 77–116; 1915–16.

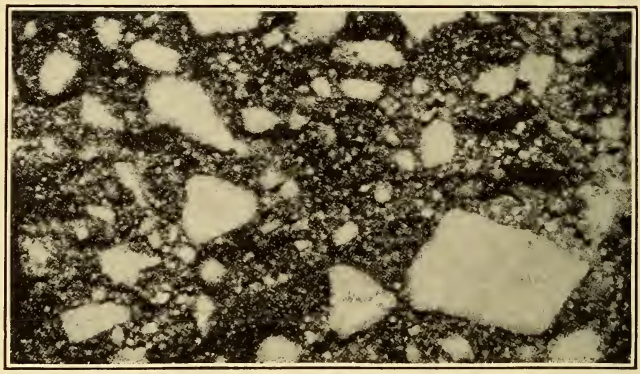

FIG. 2.—*Photomicrograph of polished surface of brick made from regular grind, × 2; Medina quartzite (after McDowell)*

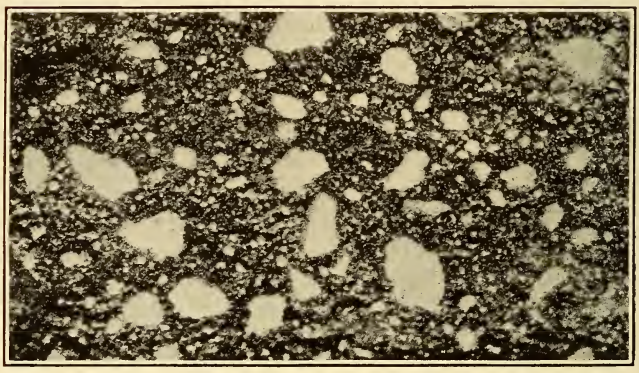

FIG. 3.—*Photomicrograph of polished surface of brick made from shape grind, × 2; Medina quartzite (after McDowell)*

of chert. As shown in this article, the flint pebbles have a lower original specific gravity than quartz, are converted to a lower specific gravity more rapidly than quartz at this temperature, and the final specific gravity is less than that of the resultant product from quartz.

In connection with load tests on silica brick, the work of Desher and Fulweiler [8] shows that with a heating rate of approximately 450° C per hour, the specimen being tested expands decidedly during the first two hours and is then practically constant in size until a temperature of approximately 1400° C is reached, above which there is apt to be further expansion. A comparison of this high temperature expansion with the permanent expansion of the piece indicates that it (high temperature expansion) is due to permanant change rather than thermal expansion, and that in the case of underburned material it will be considerable, while with well-burned material it will be small.

The screen analyses of the materials used by McDowell [9] in his work are shown in Table 2.

TABLE 2.—Screen Analyses of Raw Commercial Silica Brick Mixes as Given by McDowell

Grind	Percentage held on screen					Per cent through 40-mesh
	8-mesh	16-mesh	20-mesh	30-mesh	40-mesh	
Regular	15.3	13.4	4.6	5.4	4.8	56.5
Medium or shape	10.2	13.1	2.2	5.0	4.7	64.8
Fine	----------	14.3	4.1	6.5	4.1	71.0

NOTE.—"Regular" is the grind used in making standard 9-inch bricks; "medium or shape" is the grind used in making difficult shapes; "fine" is not usually used commercially.

The specific volumes of the different forms of silica occurring in silica refractories, as given by him, are taken up below under "Nature of silica refractories." The materials used by him were made from Medina quartzite. The coarsest grind corresponds to that usually used in standard 9-inch brick. The medium grind is that which is usually employed in making shapes. The fine grind is material which, after passing through an 8-mesh sieve, was returned to the grinding pan and reground long enough to add the lime and mix it in thoroughly. Figs. 2 and 3 show sections of brick made from these grinds. These sections well illustrate the proportion of coarse to fine materials.

[8] Report of The Committee on Refractories, Am. Gas. Inst.; 1915. [9] Loc. cit.

In crushing tests he has expressed the mean compressive strength minus the average deviation as the effective ultimate strength, and likewise for transverse tests the effective ultimate strength is given as the mean modulus of rupture minus the average deviation in moduli. His data show that in compression the fine grind was the strongest and the regular grind the weakest. In the cross breaking tests the fine grind shows little if any advantage over the regular grind, while the medium grind is considerably stronger. On reburning, both compressive and transverse strengths were found to increase for all grinds until a maximum was reached in each case, after which further burnings tended to cause both compressive and transverse strength to decrease. McDowell's opinion[10] is that this decrease in strength was caused by cracks, which were occasioned by the repeated heating and cooling.

He carried out spalling tests as follows: The bricks to be tested were heated at the rate of 15° C per hour to 600° C and held at that temperature for three hours, after which they were withdrawn and placed on steel pallets away from drafts and allowed to cool. When cold they were tested and their crushing and transverse strengths were compared with those of bricks from the same lot, but which had not received this heat treatment. The results show that on repeated burning as the percentage of cristobalite in the bricks decreased and the percentage of tridymite increased the spalling tendency decreased.

Nesbitt and Bell [11] conducted a series of tests on silica brick which were specially made from Pennsylvania ganister. Three lots of rock were used which were ground dry to pass through 12, 8, and 4 mesh screens, respectively. Two per cent of lime and 9 per cent of water were mixed with the ground quartzite. These mixes were then made up into standard 9-inch brick under various pressures for each mesh. When dry the brick were given a regular silica-brick burn.

Impact, spalling, and slagging tests were made upon these brick. The impact test was conducted by placing a heated brick on end on a steel block and dropping a steel ball upon the center of the other end. The spalling test was conducted by heating a weighed brick on one end to 540° C and then plunging the heated end into water. Ten such treatments constituted a test, after which

[10] Privately communicated.

[11] C. E. Nesbitt and M. L. Bell, Proc. Am. Soc. for Testing Materials; 1917.

the bricks were dried at 100° C; all loose material removed; the remainder weighed, and the loss given in per cent of the original weight. The slagging test was conducted by heating a brick, containing a cavity of known cross section, to 1350° C in a furnace and placing in the cavity a definite amount of slag of known composition. The temperature was held at 1350° C for two hours from the time the slag was added, after which the furnace was permitted to cool. When cold the brick was sawn in two through the center of the cavity. By means of a planimeter the area of the slag penetration was measured, and from this was subtracted the cross sectional area of the original cavity. Nesbitt and Bell's figures indicate that increased pressure in molding, between 187 and 2500 pounds per square inch, is accompanied by a continuous increase in resistance to impact; that slag penetration increases with increased size of grain; and that the spalling tendency decreases with increased size of grain. In summing up their work they conclude that as a whole the coarsest grained material used by them gave the best results.

TABLE 3.—Crushing Strengths of "Star" Silica Brick at Various Temperatures

Temperature in degrees centigrade	Crushing strength of hot brick		Temperature in degrees centigrade	Crushing strength of hot brick	
	Kilograms per square centimeter	Pounds per square inch		Kilograms per square centimeter	Pounds per square inch
15	170	2418	1200	85	1209
520	158	2247	1320	62	882
670	150	2133	1460	50	711
800	139	1977	1540	37	526
950	125	1778	1600	30	427
1050	120	1707	1700	12	[a]171

[a] Extrapolated.

In Table 3 are presented data of crushing tests on Star Silica brick at various temperatures, as reported by Le Chatelier.[12] The 1700° C figure has been extrapolated by a continuation of the curve.

[12] Revue de Metallurgie; June, 1917.

III. GENERAL RÉSUMÉ OF DATA RELATING TO MATERIALS, MANUFACTURE, AND USES

1. NATURE OF SILICA REFRACTORIES

It is evident that silica refractories must act primarily as does SiO_2. The stability relations of the different crystal modifications of SiO_2 as determined by the Geophysical Laboratory of the Carnegie Institution, briefly summarized are:

Inversion	Temperature	Remarks
α quartz—β quartz	575° C	Rapid, reversible.
β quartz—β_2 tridymite	870° C±10° C	Very sluggish, reversible.
β_2 tridymite—β cristobalite	1470° C±10° C	Do.
α tridymite—β_1 tridymite	117° C	Rapid, reversible.
β_1 tridymite—β_2 tridymite	163° C	Do.
α cristobalite—β cristobalite	220–275° C[a]	Do.

[a] Temperature depends on previous heat treatment; probably monotropic. Changes slowly to chalcedony, tridymite, or quartz in the presence of a flux at relatively low tempertures.

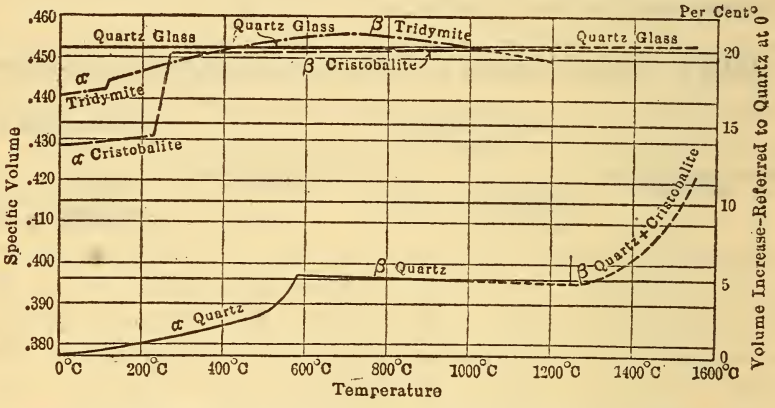

FIG. 4.—*Specific volumes of the silica minerals and quartz glass (arranged by McDowell).*

Cristobalite was formerly believed to melt to a silica glass at approximately 1625° C. More recent investigations have shown that its melting temperature under certain conditions is considerably above this figure.

Silica crystallizing from molten silicate magmas forms practically pure crystals, except that they may contain very small quantities of calcium silicate in solid solution.

"If either amorphous modification (silica glass or precipitated silica) is heated without a flux at 1300 ° C or 1400° C cristobalite

alone is obtained, although the temperature is within the range of tridymite. The process halts at the cristobalite stage, and can only be carried to completion by the addition of a flux. Similarly, ground quartz heated without a flux at high temperatures but still below the 1470° C inversion point is changed to cristobalite and not tridymite which might be expected." [13] A large pure quartz crystal when similarly heated without a flux will be converted on the surface and along cracks to cristobalite, which on cooling forms a white coating over the clear unaltered quartz of the crystal, and tends to spall off because of the Alpha-Beta cristobalite inversion.

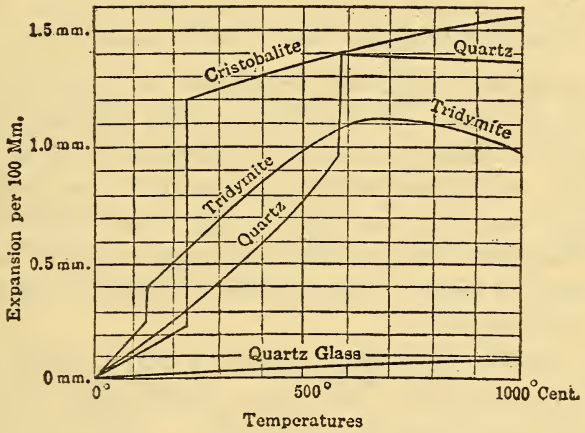

FIG. 5.—*Results of Le Chatelier's experiments in thermal expansion.* [a]

In Figs. 4 and 5 are shown the specific volumes of the different silica minerals and of silica glass and the thermal expansion of these minerals according to Le Chetelier's experiments. Results given by McDowell and others on the quantitative microscopic determinations of the various crystal forms of silica in silica refractories made from quartzites, indicate that the inversion of quartz to cristobalite takes place, but that with long continued heating tridymite eventually makes its appearance after a large percentage of the quartz has already been converted into cristobalite. Table 4 shows the micro-analyses of brick made from Medina quartzite which were repeatedly burned in a commercial kiln to cone 14–15.

[a] Le Chatelier; La Silica, Revue, 1, p. 90; 1913.
[13] C. N. Fenner, Stability Relations of The Silica Minerals, Am. Jour. Soc. 36, p. 339.

TABLE 4.—Percentages of Quartz Plus Silicates, Cristobalite, and Tridymite present in Silica Brick Made from Medina Quartzite after Repeated Heatings in a Commercial Kiln to Cones 14–15

Number of burn	Percentage by volume		
	Quartz plus silicates	Cristobalite	Tridymite
1	25	71	4
3	14	64	20
4	12	58	30
6	14	42	44
8	14	33	53
10	11	28	61

NOTE.—Results on burns Nos. 6, 8, and 10 privately communicated by McDowell.

The figures well illustrate the change of quartz through cristobalite to tridymite. The indications are that for this material and method of heating the final equilibrium would be as follows: The quartz + silicates would decrease to 13 per cent, at which point there would be no quartz left. The cristobalite would entirely disappear and the tridymite content would hence rise to 87 per cent as a limit. The material of burn No. 10 is the same as No. 6 of Table 16.

In the system CaO–SiO_2,[14] where a large excess of SiO_2 is present, as is the case with silica refractories, the lime-silica compound formed is probably $CaOSiO_2$. α $CaOSiO_2$ is the stable form from 1200° C to its melting temperature 1540° ± 2°C. Below 1200° C β $CaOSiO_2$ is the stable form. This inversion at 1200° C may possibly have a slight tendency to disrupt the ware, but the quantity of this material present, if any, is so small that it can hardly be expected to have any pronounced effect. The eutectic between $CaOSiO_2$ and silica has the weight percentage composition, CaO 37, SiO_2 63; its melting temperature is 1436° ± 2° C. In the neighborhood of pure SiO_2, however, and for some 8 to 10 per cent by weight composition from pure silica the melting temperature does not drop much below 1600° C.

In the system Al_2O_3–SiO_2 the low eutectic has the weight percentage composition Al_2O_3 13, SiO_2 87 and melts at 1610° C. In the neighborhood of pure silica, however, and up to the limits of Al_2O_3 (0.5 per cent) occurring in silica refractories, the melting temperature is close to that of pure SiO_2. In the ternary system CaO–Al_2O_3–SiO_2 the region near pure SiO_2 shows that for the

[14] Data taken from Am. J. S., 39; January, 1915. The Ternary System CaO–Al_2O_3–SiO_2.

percentages of CaO and Al_2O_3 under consideration, the melting point gradually increases as we pass from CaO to Al_2O_3.

Data of the Geophysical Laboratory published and unpublished indicate that iron oxide which may be scattered over the surface of the grains of a quartzite as hydrated oxide, gathers into grains of hematite (Fe_2O_3) when heated to comparatively low temperatures. The hematite so formed dissociates slightly, even at 1100° C and under the oxygen pressure of the air (152 mm of mercury), forming a solid solution of magnetite (Fe_3O_4) in hematite. As the temperature is raised more oxygen is evolved from this solid solution and finally the composition of Fe_3O_4 is reached at 1580° C, at which temperature melting of pure magnetite takes place. These grains of magnetite may combine with adjacent silica grains, if subjected to a reducing atmosphere, to form local spots of iron-bearing silica glass. Other ingredients, such as CaO for instance, in conjunction with the iron oxide, may tend to cause softening of the quartzite at a comparatively low temperature. It has been shown that the eutectics (points of maximum fusibility) in the system CaO–Fe_2O_3 form exceedingly fluid liquids and are at approximately 1200° C, and consequently the effect of a few per cent of iron oxide and lime on the softening of a brick may be very great. This effect may be minimized by other factors, such as lack of contact, etc. With pure silica in contact with iron oxide the effect is more difficult to predict, as this system has not been worked out. The data recorded below indicate, however, that the effect is much less than is ordinarily supposed.

The work of Fieldner [15] and others in connection with the effect of iron oxide upon the fusion temperatures of kaolin + CaO, and coal ash + CaO indicates that up to approximately 5 per cent Fe_2O_3 the iron had but little effect in reducing the softening temperatures of the mixtures, and that for this amount of iron there was very little difference in the fusion temperatures whether oxidizing or reducing conditions prevailed.

The most refractory silica brick when made into cones and heated rapidly, as described later, soften and bend over at a slightly higher temperature than that at which Orton cone 32 bends over. Kanolt[16] says: "pure silica melts at about 1600° C.[17] However,

[15] Technologic Paper No. 154, U. S. Bureau of Mines, as yet unpublished, Effect of Iron Compounds on the Softening Temperature of Coal Ash, Kaolin, and Kaolin-Lime Mixtures, in various atmospheres.

[16] Technologic Paper No. 10, this Bureau, p. 14, Melting Points of Fire Brick.

[17] This temperature was later corrected to approximately 1625° C as given above, and more recently to 1715±10° C.

the fused silica possesses such extreme viscosity near the melting point that it does not flow or change shape distinctly until considerably higher temperatures are reached. I have obtained 1750° C as the apparent melting point of pure silica; i. e., the temperature at which it flows distinctly. This temperature, however, is naturally a very indefinite one. The temperature at which silica brick flow distinctly is more definite.

2. GEOLOGY

The matter presented under this heading is intended to indicate what some of the chief varieties of material are, which have been used for silica refractories, and in general how they occur. For as new deposits are opened up it is probable that their value can be more or less predicted by such knowledge.

It is probable that most silica brick in all countries have been manufactured from the older quartzites. Although amorphous silica, chert, chalcedony, vein quartz, and quartz schist have been used to some extent, Khoetsky [18] suggests that the order of merit for silica refractories is chalcedony, old quartzites, and vein quartz, and also that quartz schist, sandstone, and sand are unsuitable for the manufacture of brick, the first on account of its structure and the presence of many impurities in the form of inclusions; the two latter on account of their variability of composition and their excessive fineness after being ground.

In the United States quartzite is the usual material, although on a small scale silica sand cemented with chalcedony and minute quartz crystals, and a pure rock chert are used, and in one case a small percentage of a material consisting of quartz pebbles cemented together by chalcedony is introduced into the brick mix.

Quartzites are sedimentary deposits and consist of silica sand, cemented together by silica. In some cases the grains still show a rounded water-worn form, while in more highly metamorphosed varieties the additional precipitated silica has built up the grains so that they are angular and tightly interlocking, thus forming a rock of low porosity. The metamorphosis in most cases is probably caused both by heat and by the deposition of silica from percolating waters containing it. Quartz schist results from quartzite when the alteration or metamorphosis is accompanied by mechanical deformation, by which a cleavage or schistose structure is developed.

[18] Khoetsky, A. K., Rev. Soc. Russe de Metall, 1, pp. 170–202; 1914.

Chert is sedimentary and is laid down from siliceous sponges, etc. It is frequently found interbedded with limestone. It is cryptocrystalline, showing slight crystal formation in a mass of amorphous material. It is evidently amorphous when first laid down. Probably some of it is opaline, as there usually appear to be small amounts of water combined with the silica.

Chalcedony may be laid down from water solutions at comparatively low temperatures, even down to atmospheric temperature. Quartz crystals also frequently form, admixed with the chalcedony.

With reference to the sources of silica brick materials in Great Britain, the following statements are taken from a paper by Bywater:[19]

The principal sources of this (silica brick material) are in the neighborhood of Neath Valley in the south of Wales, and the "black" or quartz ganister found in the neighborhood of Sheffield; * * * they have very similar composition. Samples of raw material have been found to contain:

	Dinas quartz	Black Ganister		Dinas quartz	Black Ganister
SiO_2	97.6	98.5	CaO	0.2	0.2
Al_2O_3	.5	.3	K_2O	.09	Trace
Fe_2O_3	1.5	1.3	Na_2O	.03	Trace

The South Wales or Dinas quartz has a pale grayish blue tinge and is worked in open quarries about 50 to 60 feet high with thin veins of yellow clay between. The rock is hard, and steam or compressed air drills are used. Very little impurity is found, and the rock is crushed and mixed with 1 to 1.5 per cent of lime and a small percentage of siliceous clay to toughen the brick. * * * No calcined material is used. The brick are burned at about 2600° F (1427° C) and expand somewhat on burning and subsequent heating.

"Black" Ganister.—This forms the seat stone (floor) beneath certain seams in the lower coal measures near Sheffield. It is mined by means of rock drills; and the method of manufacture is similar to that of the Dinas quartz brick, except that there are some fairly well-defined impurities found in the raw material which can be picked out. There is also usually an admixture of 25 per cent of calcined material.

The Millstone grit formation, which lies below the coal measures of the carboniferous age occurs in quantity near Sheffield. In some cases blocks of this have been used in furnaces with fair results.

Over half the rock used in the United States for silica refractories is derived from the Tuscarora (more popularly known as Medina) formation, and comes almost entirely from Blair and Huntingdon Counties, Pa. This formation consists of two distinct divisions and is immediately underlain by the Juniata forma-

[19] Frederick J. Bywater, reprint in Trans. of The Faraday Society, 12; June, 1917.

tion. (The Oneida of the Second Geological Survey of Pennsylvania.)

In the Second Geological Survey of Pennsylvania, these three formations are considered under one heading. The uppermost layer or white Medina is made up chiefly of massive beds of hard white and greenish-gray flinty sandstone; fine grained, compact, homogeneous, and in most parts of these two counties contains few pebbles.

The lower division or red Medina consists of an upper portion of red sand rocks and a lower portion of alternate layers of sandstones and shales. The Oneida consists of gray ochre pitted sandstones, the lower strata of which are quite hard.

The combined thickness of the Medina and Oneida frequently varies from 1000 to 2000 feet. The upper or white Medina usually consists of approximately half of the total. The other two are frequently of equal thickness but vary greatly.

In some places these beds lie nearly horizontal and at others are inclined at various angles. The hard white Medina forms the tops of the ridges. Lower down, the red Medina, which is softer, has weathered away and still lower, the Oneida, which is harder than the red Medina but softer than the white, projects and forms terraces along the sides of the hills. As the red Medina weathers away the hard white material breaks off and forms extensive ganister floes ranging from a few to many feet in thickness, such as are shown in Figs. 6 to 10, inclusive. This talus rock is the material usually used in the manufacture of silica brick, but in some cases it is quarried from the solid formation as is shown in Fig. 11. In the neighborhood of this quarry the white and red formations have approximately the following composition:

	White	Red		White	Red
	Per cent	Per cent		Per cent	Per cent
Loss on ignition	0.15	0.42	MgO	0.02	0.03
SiO_2	99.17	97.77	TiO_2	.02	.07
Fe_2O_3	.06	.36	Alkali	.13	.21
Al_2O_3	.45	1.20			
CaO	.02	.02		100.02	100.08

A quartzite which is quarried in Lehigh County, Pa., for silica brick purposes is also probably white Medina.

Small percentages of the red Medina are sometimes used in conjunction with the white, and in some places some of the purer portions of the Oneida formation are used. The glass sand quarry shown in the foreground of Fig. 10 is in Oriskany quartzite. For

FIG. 6.—*Medina ganister floe, Juniata River, near Mount Union, Huntingdon County, Pa.*

FIG. 7.—*Medina ganister floe, north side of Juniata River, near Mount Union, Huntingdon County, Pa.*

FIG. 8.—*Outcrop of Medina, top of Jack's Mountain, Huntingdon County, Pa.*

FIG. 9.—*Ganister talus slope on Jack's Mountain, formed from Medina outcrop shown in Fig. 10, near Mapleton, Huntingdon County, Pa.*

FIG. 10.—*Medina ridge in background, showing talus slope. Pennsylvania Glass Sand Co. Quarry (Oriskany formation) in foreground. Looking northeast along strike, Mapleton, Huntingdon County, Pa.*

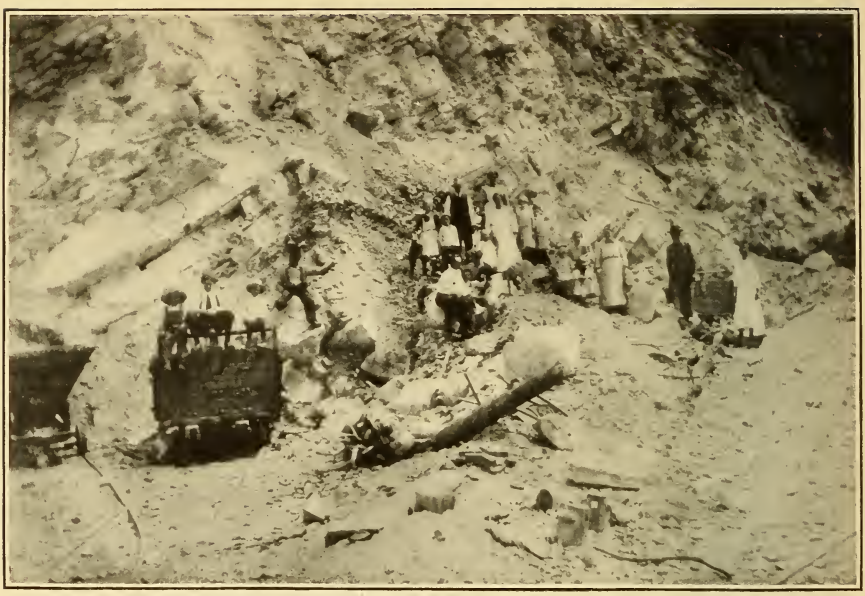

FIG. 11.—*Medina quarry, showing dip of strata, Brooks Mills, Blair County, Pa.*

FIG. 12.—*Homewood quartzite quarry, line of separation between upper and lower beds is horizontal and 3 feet below drill set up, Layton, Fayette County, Pa.*

glass sand purposes the loose grained weathered material from strata which are on edge is much sought after. However, there are localities where the Oriskany is a hard, firmly bonded quartzite of good purity. In such places it should be suitable for the manufacture of silica brick.

In Fayette County, Pa., the homewood sandstone formation of the Pottsville series is used to some extent. It has not been metamorphosed quite as much as most quartzites which are used for silica refractories, and hence is slightly softer. It is usually quarried as shown in Fig. 12. It usually occurs in two well defined layers. The upper is white and large grained, and at times has been used as glass sand. The lower one is finer grained, gray and less pure. In the view the line of separation between the two is approximately 3 feet below the drill set up. The composition of the two strata in the neighborhood of this quarry are:

	Upper	Lower		Upper	Lower
	Per cent	Per cent		Per cent	Per cent
Loss on ignition	0.30	0.50	MgO	0.03	0.05
SiO_2	98.82	97.44	TiO_2	.03	.13
Fe_2O_3	.06	.17	Alkali	.11	.19
Al_2O_3	.60	1.57			
CaO	.02	.02		99.97	100.07

In the manufacture of silica brick these two materials are frequently mixed together in equal proportions.

Wisconsin probably ranks second in the production of quartzite for silica refractories. A typical analysis of this material is as follows:[20]

	Per cent
SiO_2	97.15
Al_2O_3	1.00
Fe_2O_3	1.05
CaO	.10
MgO	.25
Alkali	.10
	99.65

Here the rock all comes from the Baraboo formation of the Devils Lake region. This formation which is a true sedimentary quartzite, consists of a series of bluffs or ranges several miles long and wide and several hundred feet high. The upper portion which forms the great bulk of the formation and is the part used for silica refractories, consists of medium-sized, rounded grains

[20] K. Seaver, Am. Inst. Min. Engrs., Trans., 53, pp. 127-128.

of quartz cemented together by secondary deposition of quartz, forming a tightly interlocking mass. Its color is generally pink or shades off into purple or gray. Only in a few places is it white.

Scattered through the massive quartzite are layers of quartz schist varying in thickness from a few inches to several feet. These are parallel to the bedding planes and have been found to have originally contained slightly more clay than the rest of the material. At the bottom of the formation the quartzite gives way to a highly siliceous conglomerate. Quarrying has been conducted chiefly at two places—Devils Lake, which is located on what is known as the South Range, and at Ablemans, which is on the North Range. Crushers are usually located near the foot of the bluffs. Hence detached fragments scattered over the hillsides may be used or the material may be blasted off the solid. In locating quarries in this region care is usually taken to see that the rock contains a sufficiently high percentage of silica, and that the quarry site is free from certain strata, which occur throughout these deposits and which are considered objectionable. A considerable part of the material used from this region thus far for silica brick has consisted of boulders collected from the surface.

The two following districts are described to give an idea of the variety of formation from which rock for silica refractories may be obtained.

In the region covered by the Pueblo (Colo.) Folio of the United States Geological Survey, the Dakota formation, from which rock for silica refractories is taken, is practically all quartz sandstones interlaid with thin shale beds. Its total thickness is usually several hundred feet. The beds vary greatly from place to place and tend to be more porous in the lower portion than near the top. In this district the Dakota lies unconformably upon Juratrias beds, thus omitting the Commanchean. At the top the Dakota passes gradually into shales of the next cretaceous formation without any sharp line of separation. Toward the top the shaly members of the Dakota become gradually more numerous and the sandy members thinner until the latter cease altogether. The highest sandstone is usually a single layer of dense, brittle rock having a vertical fracture. In these upper layers shales suitable for fire clay purposes occur. The sandstones are usually light gray, pink or white, weathering at the surface to yellow, orange, and brown. They vary considerably in friability; therefore it is necessary to exercise care and select only firmly bonded materials.

In Montana material for silica refractories is obtained from the Quadrant formation which is described as follows in the Philipsburg (Mont.) Folio of the United States Geological Survey. In this district the Quadrant formation is the only representative of the Pennsylvanian series. It rests unconformably upon the Mississippian series and is overlain unconformably by the Jurassic. It consists of two members. The lower one is a series of red magnesian limestones and shales. The upper member, mainly quartzite, comprises three strata, the lowest consisting of light-colored, pure, thick-bedded quartzite; the middle of calcareous shale and impure cherty limestone; and the uppermost of somewhat impure quartzite and quartzitic sandstone. The lower massive quartzite is apparently 350 feet thick in places, and is probably the more valuable rock for silica refractories because of its purity and dense structure, although the upper beds are also of considerable thickness and may furnish suitable material at certain places.

The material used in the manufacture of silica brick in Alabama is a hard quartzite with tightly interlocking grains. It occurs as loose boulders which are present in large quantities and appear to be from one of the older formations, probably the Weisner formation of Lower Cambrian age.

The geologic ages of the various materials used for the manufacture of silica brick in the United States are shown in part in Table 5.

TABLE 5.—Geologic Column with Reference to Commercial Quartzites for Silica Refractories

Major divisions				Materials used commercially
CENOZOIC ERA				
Quaternary	Pleistocene.			
Tertiary	Pliocene.			
	Miocene.			
	Oligocene.			
	Eocene.			
MESOZOIC ERA				
Cretaceous	Laramie.			
	Montana..	Western Interior.		
	Colorado..			
	Dakota....			
Comanchian	Washita		From these come...	Colorado quartzites.
	Fredericksburg..	Western Gulf Region..		
	Trinity.....			
Jurassic.				
Triassic.				

TABLE 5.—Geologic Column with Reference to Commercial Quartzites for Silica Refractories—Continued

PALEOZOIC ERA

Major divisions				Materials used commercially
Permian	Dunkard.			
Pennsylvanian (Upper Carboniferous)	Coal Measures	Monongahela	Upper productive.	
		Conemaugh	Lower barren.	
		Allegheny	Lower productive.	
	Pottsville (Millstone grit)	Homewood sandstone	Quadrant Formation of Western Montana	Homewood sandstone, Pennsylvania. Montana quartzite.
		Mercer shale, coal, and fire clay		
		Connoquenessing		
		Sharon shale and coal		
		Sharon or Olean conglomerate.		
Mississippian (Subcarboniferous)	Kaskaska (Chester)			
	St. Louis	Mississippi River States	Mitchell Limestone Formation	Indiana chert.
	Osage (Augusta)			
	Kinderhook			
Devonian	Upper	Chautauquan (Chemung–Catskill groups).		
		Senecan	Portage–Naples–Ithaca groups.	
	Middle		Genesee–Tully groups.	
		Erian	Hamilton group.	
			Onondaga group.	
	Lower	Oriskanian (Esopus–Oriskany groups).		
		Helderbergian (Becraft – New Scotland – Coeymans Formations).		
Silurian	Upper (Cayugan)	Monroe group. (Manlius the youngest formation.)		
		Salina group.		
	Middle (Niagaran)	Guelph dolomite	Louisville limestone.	
		Lockport dolomite		
		Rochester shale		
		Clinton Formation.		
	Lower (Oswegan)	Medina	White Medina	Medina quartzite, Pennsylvania.
			Red Medina	
		Oneida		Oneida quartzite, Pennsylvania.
Ordovician	Upper (Cincinnatian)	Gamache.		
		Richmond.		
		Maysville and Lorraine.		
		Eden and Utica.		
	Middle (Champlainian)	Collingwood.		
		Trenton.		
		Black River.		
		Lowville.		
		Chazy and Stones River, St. Peter.		
	Lower (Canadian)	Fort Cassin.		
		Beekmantown.		
Cambrian	Upper or Potsdam.			
	Middle or Arcadian.			
	Lower or Georgian. (Probably Weisner Formation			Alabama quartzite.

PROTEROZOIC (ALGONKIAN)

Keweenawan.				
Huronian	Upper.			
	Middle (Baraboo formation, probably Middle Huronian)			Wisconsin quartzite.
	Lower.			

ARCHEOZOIC (ARCHAEAN)

An inspection of this table indicates what a great variation there is in the various materials in this respect.

3. MANUFACTURE

The following remarks describe the general method of manufacture at a plant which uses Pennsylvania Medina quartzite

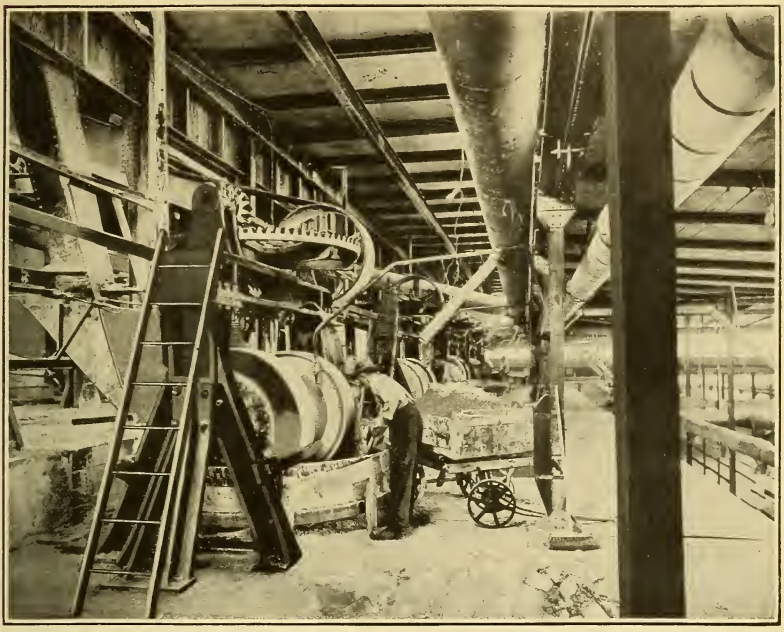

FIG. 13.—*Wet pan for grinding silica brick mix, showing tube used for adding milk of lime (Harbison & Walker plant)*

FIG. 14.—*Showing method of molding silica brick by hand, and method of placing on dryer cars (Harbison & Walker plant)*

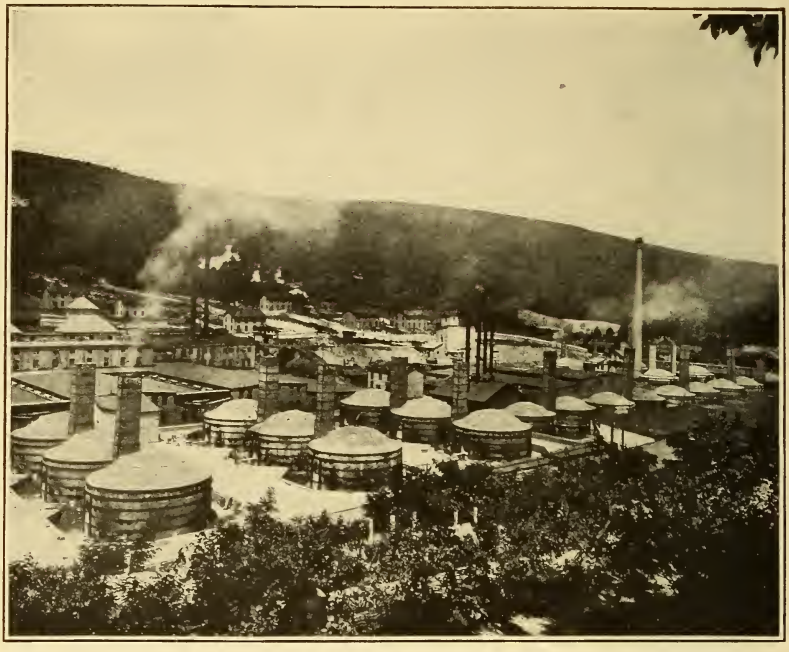

FIG. 15.—*Kilns for burning silica brick, showing holes in crowns of kilns for cooling, and ganister floes in distance (Harbison & Walker plant)*

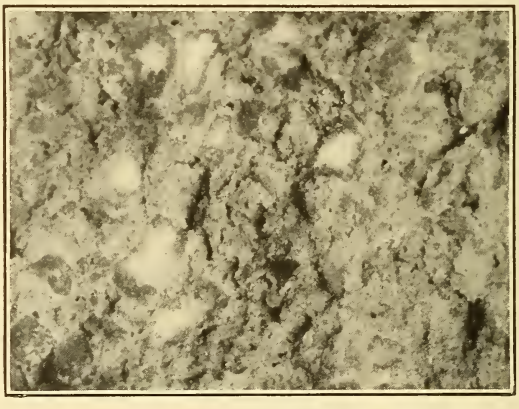

FIG. 22.—*Brick made from Medina quartzite, after 40 heatings to 1450° C; groundmass is tridymite, gray outer portion of large particles is tridymite in glass, and white centers of large particles are cristobalite (natural size)*

Most other silica brick plants in the United States follow approximately this procedure except as noted below.

The rock direct from the quarry is crushed to pass through a 2-inch ring. Washing of the rock to remove foreign or earthy matter may be resorted to at certain places. The material is then taken in a car to the wet pan and there dumped. Each charge for a pan weighs about 1250 pounds. A little water is added and the material is ground for 15 minutes. After 10 minutes of this grinding, 2 per cent of lime by weight is added as slaked high-calcium lime, in the form of a thin slip. It is run through a spout to the middle of the crushing surface of the pan, while the pan is in motion. This is well shown in Fig. 13. In this way the lime is thoroughly mixed with the quartzite. When the grinding has continued for the full 15 minutes the mix is ready for molding and is removed from the pan to a tramcar by means of a mechanical scraper.

The lime is prepared by weighing out enough quicklime to furnish exactly 2 per cent of CaO to the weight of quartzite. The weighing out is done on the basis of an analysis. Thus, if the lime contains only 90 per cent CaO, proportionately more is added. This lime is then placed in a small mixer or blunger; is slaked and stirred up to the proper consistency; and then run through a screen of approximately 10 meshes per linear inch on its way to the wet pan. Hydrated lime is sometimes weighed into the mixer instead of quicklime and appears to give exactly the same results.

From the pan the mix is carried to an elevator, raised to an upper floor, and dropped into a container, from which the workmen use it directly in filling the brick molds. This container is in the nature of a cylinder approximately 5 feet in diameter and 10 to 15 feet tall, with slatted openings at the bottom, so that the material can be removed readily. The slats can also be removed. The molders work at benches adjoining the outlets of the containers. The brick molds are of iron and consist of a bottom plate on which lies a 3-brick form. The brand on the brick is formed by the bottom plate of the mold. Directly above the molds is situated a screen which can be shaken by the operator. Pennsylvania washed glass sand is placed upon this screen and a slight jar is sufficient to shake down enough sand to sand a set of molds.

As shown in Fig. 14, a mass of mix is wadded together with the hands and is then thrown heavily into the molds and rammed in,

the surplus struck off and the bricks turned out onto an iron pallet. These pallets are then placed on drier cars. When a car is full it is left standing in the open for a few minutes and is then run into a hot-air drier. Regular burn brick are made up just wet enough so that they will fill the corners of the molds readily. The mix for large shapes is made slightly drier than for regular brick, but the grind is longer, approximately two or three pans per hour instead of four. (The grind for regular brick is at the rate of four pans per hour.) This extra grinding aids in producing a more plastic consistency. The material for large shapes is pounded into the molds with heavy wooden mallets. As the jars due to the plant engines and passing trains tend to cause large shapes to deform before they get their initial set, the stiffer they are when they come from the mold the better.

Of course the time of grinding at each plant will depend somewhat upon the nature of the quartzite used and the ware being turned out. Some plants make up brick as described above, and when the bricks are partially dry they are re-pressed. Another way is to have the mix somewhat drier to start with and make the brick up directly on a dry press machine.

Practically all types of driers used for drying heavy clay wares have also been applied to drying silica wares. Perhaps the most satisfactory type from an economical standpoint is a Richardson type tunnel drier, using waste heat from cooling kilns, and if necessary an auxiliary heater, either a small furnace and brick checkerwork, or else by passing air over exhaust steam pipes. Silica brick give practically no trouble in drying. The only requirement is that the brick must be bone dry before being set in the kilns. Otherwise, they break under the pressure when set, or tend to slump and break during water smoking.

As shown in Fig. 15, the kilns are of the regulation circular down-draft type, varying from 30 to 40 feet in diameter. The bag walls are usually low, as direct fire does not appear to affect the brick much. Regular 9-inch silica brick are set approximately 3 to 3½ on 1 or, in other words, ½ inch is left between the bricks to allow for expansion. They are put in benches 4 feet wide by the width of the kiln by the height of the kiln and a space of 4 inches is left between benches, and approximately 18 inches between the top of the bench and the crown of the kiln. This latter is done to prevent lifting of the crown of the kiln by the expanding brick. Thus, the benches are from 12 to 15 feet high. As set in the kilns there is a pressure of approximately 15 pounds

per square inch on the bottom brick, which do not crush or crack if they are thoroughly dry. A course of bats is put in occasionally to level up the bench horizontally, but they are not used in any way to take the load off the unburned brick. In general the time of burning is nine days with a maximum heat treatment equivalent to cone 14–18. Five to eight additional days are required for cooling.

A departure from the usual method of burning, which is used by some plants, is the continuous-tunnel kiln. With these kilns the period of burning is approximately a week, and the cooling takes another week. The maximum heat treatment is equivalent to cone 12–14.

In England and other European countries brick which contain 96 per cent or more of SiO_2 are manufactured in a manner similar to that used for making silica brick in the United States, except that the methods of mixing, etc., vary somewhat. It is a frequent custom in England to use a somewhat lower percentage of lime (1.3 per cent or even less) and add a small percentage of a highly siliceous clay to strengthen the brick in the raw state. If clay were not added to these brick, they would be weak because of their low lime content.

In England segmental gas retorts are frequently made from materials which contain 85 to 92 per cent of SiO_2. Such pieces are, of course, made by mixing larger percentages of siliceous clays with the silica, and hence, do not correspond to silica brick as manufactured in the United States.

4. USES IN INDUSTRIES

Silica refractories are particularly useful in industrial work. This is due to their rigidity at temperatures above the range of usefulness of ordinary fire-clay brick, and to the fact that they expand slightly when heated instead of shrinking as clay brick do.

For by-product coke-oven work the brick in the regenerative and lower two-thirds of the coking chamber should have a high fusion point, as an extreme temperature of 1510 to 1540° C is sometimes reached. The brick in the lower two-thirds of the coking chamber, where they come in contact with the coal and coke during charging and unloading must also resist abrasion. The upper one-third of the coking chamber may be made of lower fusion point material and need not be particularly resistant to abrasion, as here the temperature rarely exceeds 1320° C and the brick are seldom touched by coal or coke. Occasionally charges of coal have been

found to expand in the coking chambers, so that when coked they could not be removed by the usual mechanical device. Trouble from cracking and spalling may sometimes occur if the ovens are heated up too rapidly. However, when the temperature of a series of these ovens is once raised to the operating temperature, they should last for years, except for repairs occasioned by abrasion, etc.

From the work of Blasberg [21] we gather that silica refractories which have been used in Siemen's by-product coke ovens frequently show an increase in iron and always a considerable increase in alkalies, particularly the brick used in the checkerwork. In many cases they contain up to 1 per cent of carbon. It is assumed that the carbon comes from the dissociation of gaseous hydrocarbons or carbon monoxide, and that the other contamination comes from the coal ash. The conclusions drawn are that for this work silica brick should be as low in porosity as possible and that the gases should be free from flue dust.

In open-hearth steel furnace work, silica brick are used for the top, ends, and sometimes for the side walls of the furnace. Here the chief essentials are that the brick have a high crushing strength when hot and high resistance to spalling and slagging, as well as high softening temperature.

The port bulkheads at the ends of these furnaces are subjected to a severe sand-blast action from particles of limestone and iron oxide, which are blown over from the hearth of the furnace. As these materials both act as active fluxes on silica brick under reducing conditions, the result is that these bulkheads are rapidly eaten away by a combined cutting and slagging action. The 13-inch brick used in these bulkheads wear away so fast that it is frequently necessary to replace them in two or three weeks. Crowns of such furnaces are also somewhat attacked by the slagging action of lime and iron oxide which are blown up against them and also by the dust of the gaseous fuel used. Thus, an average crown lasts approximately 300 heats when natural gas, which is quite free from dust, is used as a fuel; approximately 200 heats with producer gas, which contains considerable dust, and somewhat less than 200 heats when powdered coal is used.

For steel furnace work it is desirable that the softening temperature (as described below) of the brick be somewhat above that of Orton cone No. 31. Even then, at times the surface of the bricks

[21] Stahl, W. Eisen, 30, pp. 1055–1060; 1910.

will melt and hang down in long white stalactite like forms from the crown of a furnace.

The uses of silica refractories in copper reverberatory furnaces are similar to those in open-hearth steel furnaces, except that for melts of lead-free metallic copper, silica can be used throughout the furnace except at the slag line. For this work the brick should be as low in porosity as possible to avoid absorption of copper oxide, which slags away the bricks when the furnace is at its maximum temperature. Silica brick arches in copper furnace work wear much better if they are not insulated. The maximum temperature reached in the copper refining furnaces is approximately 1350° C; hence, for this purpose some refractoriness might be sacrificed if anything is to be gained thereby in decreased porosity and resistance to spalling.

In glass furnace work silica refractories are used for furnace roofs, and are useful for stiffening up walls, etc., made of clay brick, which would otherwise soften and sag from the effect of heat and fumes arising from the glass.

IV. STATEMENT OF WORK DONE AND DESCRIPTION OF METHODS

1. GENERAL STATEMENT

The experimental work consisted of a number of heating tests of silica materials carried out for the purpose of determining the amount, speed, and nature of the volume changes occasioned in such materials by various heat treatments. The work includes the study of raw quartzites, raw commercial brick mixes, commercial brick, and special mixes. In addition to this the properties of finished brick and some data concerning the manufacture and qualities of materials have been considered. Wherever possible one number has been used to represent the quartzite, the burned and the unburned brick of a variety, the distinction in each case being made in the text. This applies to Tables 6, 7, 8, 12, 14, 16, 17, 18, 19, 20, and 21.

2. HEATING TESTS

(*a*) *Series 1.*—The first work carried out was to determine the exterior volume changes of a silica brick mixture on repeated burnings at successively higher temperatures. The material (No. 6) used in this first test consisted of twelve 2-inch cubes made from Medina quartzite, plus 2 per cent of lime. It was

ground and mixed in the usual way at a silica brick plant. Its screen analysis should correspond approximately to that given for regular grind (Table 2). When the cubes had been thoroughly dried at 110° C they were burned to 1150° C in approximately three days before any measurements were made upon them. After this burning the exterior volumes of the pieces were taken by means of a Seger voluminometer, using a petroleum oil as the displacing liquid. By means of a micrometer caliper, one dimension of each piece was also measured. The pieces were then burned again, this time to 1200° C, after which the volume, etc., of each piece were again measured. The next burning temperature was 1250° C. These burnings and measurements were continued at 50° C intervals up to and including 1500° C. The pieces were burned three times to 1500° C. The duration of these burns varied somewhat but the average was as follows:

Eighteen hours for heating to 800° C and six hours for heating from 800° C to the desired temperature. The pieces were held at the maximum temperature for one and one-half hours in each case. In all succeeding heating tests throughout this paper this heating procedure was followed except as noted. The volume and linear expansions were calculated in terms of the first set of measurements (those made following the 1150° C burn). Starting with the 1200° C burn one piece was saved from each burn up to and including the 1500° C burn for microanalyses.

(*b*) *Series 2.*—The above work showed such interesting results that it was decided to extend it to other varieties of material. This second test included raw quartzite, raw brick mixes and commercially burned brick. The burnings were made as with the preceding set and ranged from 1200° C to 1500° C, inclusive. The materials used in this case were two 2-inch cubes each of Medina (No. 6), Baraboo (No. 22), and Alabama (No. 26) quartzites, 12 cubes of Medina raw brick mix as used for the first test, 12 cubes of a commercial brick made from Baraboo quartzite (No. 18) and 12 cubes of a brick made from Baraboo quartzite which had been lightly calcined (No. 20) before being made into brick.

In this case the wet weight, dry weight, and suspended weight of the pieces were taken, in addition to the measurements made in the first test. These latter determinations were made by drying the pieces at 110° C, weighing dry, saturating with water and weighing wet, then weighing suspended in water which was at room temperature. Saturation was obtained by placing the pieces in an iron cylinder, covering them with boiling water, and subject-

ing them, while thus submerged, to a vacuum equivalent to 24 inches of mercury for a period of four hours. In the case of the raw brick mix it was necessary to make these weights in petroleum of a known specific gravity, for water would slake the material. The wet, dry, and suspended weight method of determining porosity, etc., was used throughout these tests, except as noted.

(*c*) *Series 3.*—The above studies indicated the desirability of knowing the effect produced in quartzite and silica brick mixes at different stages during a single burn. Hence a burn was conducted from which pieces were withdrawn from time to time. In this case the material used consisted of 42 pieces (approximately 50 g each) of Baraboo (No. 18) quartzite, and 42 two-inch cubes of a mix made by mixing 2 per cent of lime with this Baraboo quartzite that had been crushed to pass through an 8-mesh screen. The volumes, etc., of the pieces were taken, after which they were placed in a kiln in 42 small numbered clay saggers, each sagger containing one piece of quartzite, one cube, and the desired Orton cones. These saggers were covered and set two high, according to number. Heating to 1000° C took approximately two days, at which temperature box number one was withdrawn. The temperature was then raised to 1200° C and held at this temperature for one hour to insure uniformity of kiln temperature, at the end of which time box number two was withdrawn. Four more draws were made at this temperature at intervals of five hours, after which the temperature was raised to 1250° C in two hours, held for one hour, and the first 1250° C draw made. This process was continued by 50° C steps up to 1450° C, at which temperature nine draws were made instead of five, before proceeding to 1500° C, which was the highest temperature used. To prevent spalling of the materials when withdrawn from the kiln, they were cooled slowly by being placed in successively cooler kilns.

(*d*) *Series 4.*—At this juncture it was apparent that the changes taking place in commercial brick when heated once to a temperature of from 1400 to 1500° C in a manner similar to that used in series 1 and 2, were a guide to the degree of burning which the brick had received in manufacture. Following this line, several of the leading varieties of silica brick manufactured in the United States, and the corresponding quartzites and raw mixes, were tested by single heatings at temperatures of 1400, 1450, and 1500° C. Some of these heatings were carried out at the same time as those of series 2.

(*e*) *Series 5.*—Working with so many different varieties of material it was soon seen that various constituents occasionally present or added and treatments such as calcining of the quartzite before making into brick, had certain effects on the burning behavior of the wares. To determine these effects, several heats were made. Tests were carried out with iron oxide, impure ferruginous clay, pure plastic clay, sodium chloride, lime, and calcium fluoride. Loose-grained Medina (No. 37) quartzite crushed to pass through an 8-mesh screen was used in this set of experiments except as noted.

The first work consisted of two similar burns on the same pieces. The maximum temperature in each of these burns was 1400° C and the heating was as follows:

Fifty degrees C in 15 minutes from atmospheric temperature to 500° C, 75° C in 15 minutes from 500 to 800° C, 100° C in 15 minutes from 800 to 1200° C, then 50° C in 15 minutes to 1400° C and held at this maximum temperature for one and one-half hours. In this case only exterior volumes were measured. Exterior volume changes alone do not constitute a very definite indicator of the amount of transformation taking place, while as shown later, the specific gravity change in conjunction with the exterior volume and porosity changes do constitute a definite indicator of the nature of the changes taking place, and the specific gravity alone indicates the exact degree of transformation. Hence, in the balance of the work in connection with added ingredients specific gravity was used as an indicator of the degree to which the materials had been transformed.

The second test was conducted with the 1450° C burn of series 4. Hence, the figures of the present test are more or less comparable with those of the former. The third test was conducted to determine the effect of coarser grind, pressure in molding, and a slightly longer time of burning. In this case the quartzite was broken up in a jaw crusher to pass through a 4-mesh screen, and the mix was made up by hand to avoid further crushing of the material. The test pieces were molded under a pressure of 5000 pounds per square inch. In burning the temperature was raised to 800° C in approximately 18 hours, then to 1200 ° C in 4 hours, and was maintained between 1200° C and 1300° C for 2¾ days. Finally, the temperature was gradually raised to 1450° C and held at this maximum for 3 hours. A fourth test of added ingredients was conducted in connection with series 7.

For the tests of calcined materials, Baraboo (No. 18), Medina (No. 6), and loose-grained Medina (No. 37) quartzites were used. The materials in lump form were calcined twice to 1500° C, then crushed to pass through an 8-mesh sieve before being made up in a mix. The first of these tests was carried out in conjunction with test No. 2 above (this series) and the second test (the pieces for which were made up with a pressure of 5000 pounds per square inch) was in conjunction with the second burn of test No. 1 above (this series). Another test of these materials was made in conjunction with series 7.

(*f*) *Series 6.*—During the time that the above tests were being made, a number of tests of quartzites to determine their suitability for the manufacture of silica brick, were also being carried out. The first material to be tried out was the friable Medina (No. 37) described under series 5 above.

In these tests the fineness of grind, added ingredients, and method of burning were varied. The first test consisted of two mixes, one of 8-mesh material and one of 10-mesh material, each of which contained 2 per cent of CaO. Brick from these mixes were burned as in tests of series 1. The maximum temperature (1465° C) was maintained for only a few minutes. This treatment brought down cone 16. The second test was of material (No. 37) which had been previously calcined at 1300° C for 1½ hours and then quenched with water, and crushed to pass through an 8-mesh sieve. This mix was made up with 2 per cent of lime and burned the same as above, except that in this case a maximum temperature of 1485° C was maintained for a few minutes, which brought cone 17 down. The third burn consisted of mixes made up from 8-mesh material (No. 37) and 2.5 per cent lime, 3 per cent of lime, 1 per cent lime, plus 1.5 per cent Tennessee ball clay, and 2.5 per cent of Tennessee ball clay, respectively. These brick were burned same as the above to cone 16+ with a maximum temperature of 1485° C which was maintained for a few minutes. The fourth burn was for the purpose of reburning previously burned bricks and consisted of two bricks each of the mixes containing 2.5 per cent of lime, 3 per cent lime, and 2.5 per cent Tennessee ball clay, respectively, and the mix made from calcined quartzite plus 2 per cent lime. In this case the burn was similar to the preceding, except that the maximum temperature of 1450° C was maintained for five hours. The fifth test consisted of bricks made from 8 and 20 mesh materials (No. 37), respectively. In

each case 2.5 per cent of lime was used in the mix. The fifth burn also contained brick made from a strongly bonded quartzite (No. 39) having a conchoidal fracture. This rock was crushed to pass through an 8-mesh screen, and 2.5 per cent of lime was used in the mix. This burn was conducted similarly to the others, and the maximum temperature (1470° C) was maintained for 3 hours. This brought down cone 20. Brick made from commercially used Medina quartzite was tested in conjunction with these various sets of bricks.

(*g*) *Series 7.*—Satisfactory data of the relation existing between results obtained in test kilns and those obtained in regular silica brick kilns were lacking; hence, a burn was made in a commercial silica brick kiln. The materials of this test consisted of one piece of the preburned Baraboo mix described under series 5 above—shale and lime mix, sodium chloride lime mix, and a piece which contained only lime in addition to the quartzite. The materials were placed near the top of the kiln. The period of burning was 14 days, and the period of cooling was seven days. The heat treatment received by the pieces brought down cone 16+.

(*h*) *Series 8.*—This test was conducted to determine the comparative behavior of underburned, medium burned, and well-burned bricks on being rapidly reheated. The brick were made from Medina (No. 6) quartzite, and all came from the same kiln of ware. During the reheating test the temperature was raised to 1480° C in approximately six hours, and this maximum was maintained for approximately one hour.

3. PROPERTIES OF FINISHED BRICK AND DATA ON MANUFACTURE

(*a*) *Data on Commercial Bricks.*—The foregoing tests brought out the fact that the specific gravity of silica brick, as they come from the manufacturer, calculated from the wet, dry, and suspended weights, is the best guide in showing the degree of burning which the various brands receive in manufacture. Likewise, it was seen that brick made from any one variety of quartzite show certain peculiarities. It was learned from the different manufacturers that the time of burning and the cone representing the maximum of the heat treatment given in each case varied considerably. To determine the specific gravity of average burned brick the specific gravities of most of the leading varieties manufactured in the United States were taken, and to show the prop-

erties of the different quartzites these data have been arranged according to the quartzite from which the bricks were made.

(*b*) *Analyses and Softening Temperatures.*—The softening temperature of a material may be the limiting factor in its use. Hence, this point has been considered for a few special materials, and since the chemical composition of silica brick is so closely allied with the softening temperature, these two subjects are considered together.

(*c*) *Load Tests and Volume Changes at 300° C.*—Load tests have been used to some extent as a guide to the quality of silica refractories. The data here considered have to do chiefly with the rate of heating and a consideration of the volume changes in the neighborhood of 300° C, and in the neighborhood of 1400° C and above. A means of actually measuring volume changes up to 300° C is also considered.

(*d*) *Micro Studies.*—Only such micro studies have been included as are essential to the understanding of the materials used in the experiments. The work taken up includes two Baraboo quartzite draw trial pieces and the corresponding pieces of brick mix, chalcedony cemented quartz conglomerate from Colorado, chalcedony cemented quartz sandstone from Missouri, and chert from Indiana. A general consideration of the optical properties of quartzites which may aid in determining their value for silica refractories is given, as well as a few studies to show the optical properties and nature of the grind of some standard and special silica brick.

(*e*) *Effect of Slags.*—Slag tests in connection with silica refractories have received some attention, and with reference to ware for certain uses are quite important. The work here presented is principally in connection with the effect that fineness of grain has upon slagging of the brick.

(*f*) *Screen analyses.*—Screen analyses have been made of 15 representative raw brick mixes. Water was used to disintegrate the material and for washing.

(*g*) *Cold Strength Tests.*—Cross breaking tests were made of 18 varieties of commercial brick. Standard 9-inch brick were used. The brick were placed on edge on knife edges, with 6 inches between supports, as described elsewhere under cold cross breaking test.

V. RESULTS OF WORK AND DISCUSSION OF SAME

1. HEATING TESTS

(*a*) *Series 1.*—Fig. 16 shows the average percentage exterior volume increase of the pieces used for the first test. After the last 1500° C burn the average specific gravity of the five remaining pieces was 2.304. The crystal structure [22] of the cubes after each burn shows that cristobalite was present in appreciable quantities after the 1300° C burn, while tridymite did not make its appear-

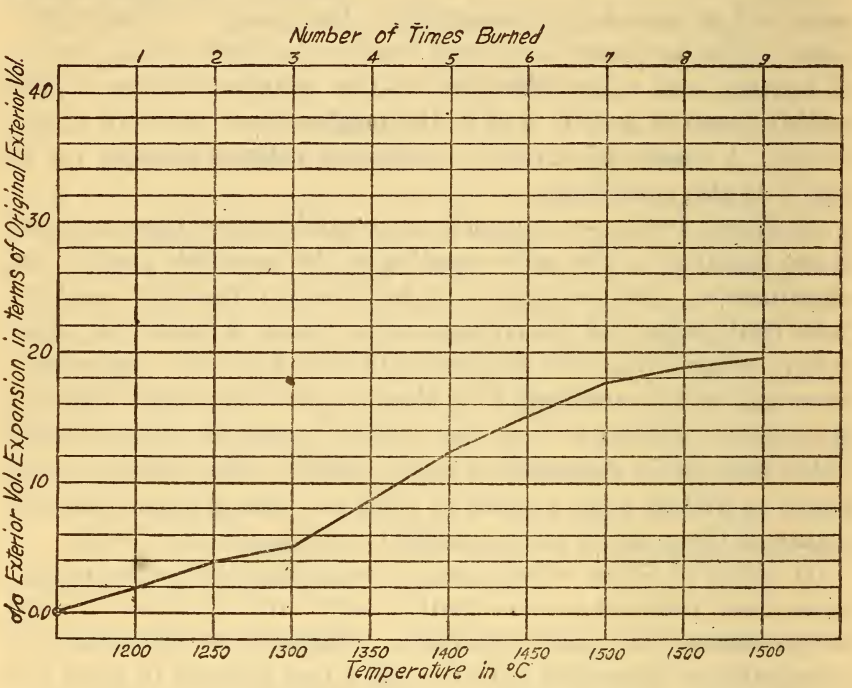

FIG. 16.—*Volume changes caused in Medina mix, which had previously been burned to 1150° C, by repeated burnings to successively higher temperatures from 1200–1500° C*

ance until the 1450° C burn, after which there was only a trace of it present, although at this stage 81 per cent of the material by volume had been converted to cristobalite. After the first 1500° C burn the tridymite had increased considerably and the amount of crystobalite had somewhat decreased. There was also a slight decrease in the amount of quartz plus silicate glass.

This series of burns although not directly comparable to a burn in a commercial kiln is nevertheless somewhat analogous, in that to a certain degree the pieces received the effect of soaking during the several burns, and each burn was carried to a higher temperature

[22] Trans. Am. Cer. Soc., 18, p. 519.

than the preceding, thus giving the effect of rising temperature. Such burning differs from commercial burning in the repetition of heating and cooling, and also in the fact that commercial burns are not usually carried to 1500° C. The curve shows the point at which further heating practically ceased to cause additional exterior volume expansion. Hence the specific gravity 2.304 approximately represents the minimum specific gravity obtainable by such heat treatments in which 1500° C is the maximum temperature reached. The crystal changes are of particular interest, in that they show that in silica brick mixes cristobalite and not tridymite is the

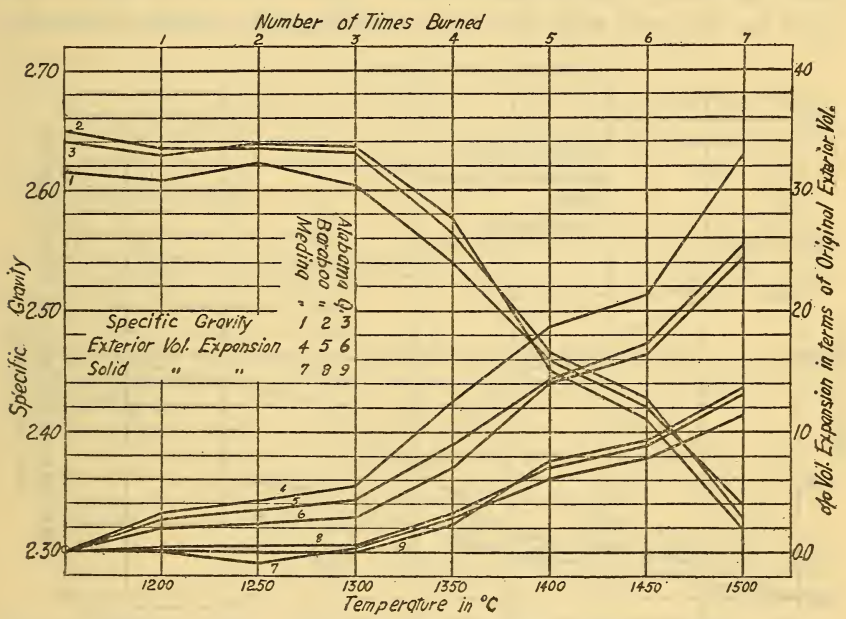

FIG. 17.—*Changes in exterior and solid volumes and specific gravities of Medina, Baraboo, and Alabama quartzites, caused by repeated burnings to successively higher temperatures from 1200–1500° C*

form that appears first and most prominently when such mixes are heated at temperatures below 1470° C. It was found that in these pieces, at temperatures above 1470° C tridymite is still present in appreciable quantities. (After third heating at 1500° C, quartz plus silicate equals 6 per cent, cristobalite 70 per cent, tridymite 24 per cent, by volume.)[23]

(*b*) *Series 2.*—Fig. 17 shows the exterior volume changes and those of the solid portions of the quartzites tested in series No. 2.

[23] Authorities differ as to whether cristobalite or tridymite is the stable form of silica at 1500° C; J. B. Ferguson and H. E. Merwin, Am. J. Sci., 4th series, 46, pp. 417–426.

These changes are figured in terms of the original exterior volumes of the pieces for the sake of convenience in comparison with the other curves. This plan is followed throughout this paper, except as noted. This figure also shows the changes in specific gravity of the pieces.

The specific gravity and solid volume curves show that up to and including 1350° C very little transformation of quartz took place, but that up to this stage there had been some increase in exterior volume of the pieces. By the end of the 1500° C burn, however, almost as much transformation had apparently taken place as would be the case with the corresponding silica brick mixes on

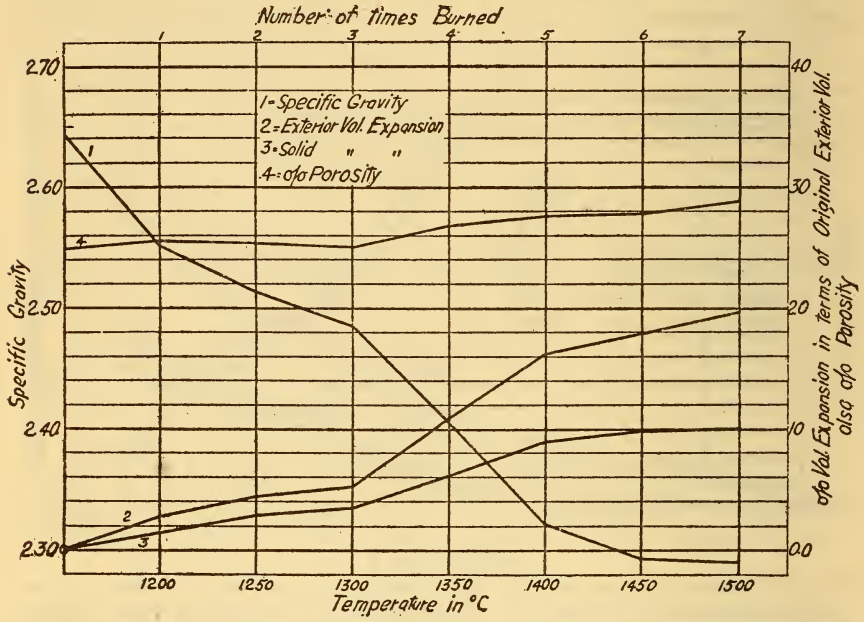

FIG. 18.—*Changes in exterior and solid volumes, specific gravity and porosity, of Medina raw mix, when heated same as quartzites of Fig. 17*

receiving similar heat treatment. It is also seen that the Alabama quartzite, which shows the greatest transformation, as indicated by the curves of specific gravity and the expansion of the solid materials, shows the least exterior volume expansion, while the Medina quartzite which shows the least transformation, shows by far the greatest exterior volume expansion. These three varieties of quartzite, which are fairly representative of the varieties usually used in the manufacture of silica refractories, do not show any great differences in their rates of transformation.

In Fig. 18 are shown the exterior and solid volume changes of the raw Medina mix, as well as its specific-gravity changes. From

consideration of the specific gravity and solid volume expansion curves it is apparent that even at temperatures as low as 1200 and 1250° C there is an appreciable amount of transformation going on in such a mix. An inspection of the exterior and solid volume curves from 1200 to 1350° C shows that there is practically no expansion of pore space during this interval, and possibly a slight contraction. In the region above 1350° C, however, where the rate of transformation increases very rapidly with increase of temperature, it is seen that the exterior volume increase is much more than that of the solid volume, thus showing that the pore space increased considerably. The final specific gravity 2.290, compared to a final specific gravity of 2.304 for the similar material of series 1, shows how close the final specific gravity of two sets of material may be when extensively heated at high temperatures, even though the methods of heating the two sets be somewhat different.

Table 6 shows the exterior volume increase and porosity change for the Baraboo commercial brick, and the brick made of lightly calcined Baraboo quartzite.

TABLE 6.—Effect on Volume and Porosity of Baraboo Quartzite Bricks, Caused by Reburning to Successively Higher Temperatures, in Test Kiln, Same as for Quartzites of Fig. 17

Material	No.	Original per cent porosity	1200° C		1250° C		1300° C		1350° C	
			Per cent volume expansion	Per cent porosity	Per cent volume expansion	Per cent porosity	Per cent volume expansion	Per cent porosity	Per cent volume expansion	Per cent porosity
Brick made from Baraboo quartzite, regular burn	18	22.26	0.190	23.70	0.438	23.36	0.440	23.67	1.731	24.51
Brick made from lightly calcined Baraboo quartzite, burned as above	20	30.09	.108	31.50	.538	31.00	.104	31.38	.412	32.41

Material	No.	Original per cent porosity	1400° C		1450° C		1500° C	
			Per cent volume expansion	Per cent porosity	Per cent volume expansion	Per cent porosity	Per cent volume expansion	Per cent porosity
Brick made from Baraboo quartzite, regular burn	18	22.26	3.408	24.73	4.36	24.80	6.49	25.99
Brick made from lightly calcined Baraboo quartzite, burned as above	20	30.09	.666	31.91	1.04	31.40	1.34	31.93

The brick made from raw quartzite increased considerably in volume and slightly in porosity, while that made from calcined quartzite shows very little increase in volume and with the lower temperatures shows a decrease in porosity. These data make it apparent that commercial brick act essentially as do raw mixes, the chief difference being the lesser amount of transformation which takes place in the commercial brick. Comparison of the data for these two varieties of brick shows what increase in the ultimate degree of burning of a brick a slight calcining of the quartzite will produce.

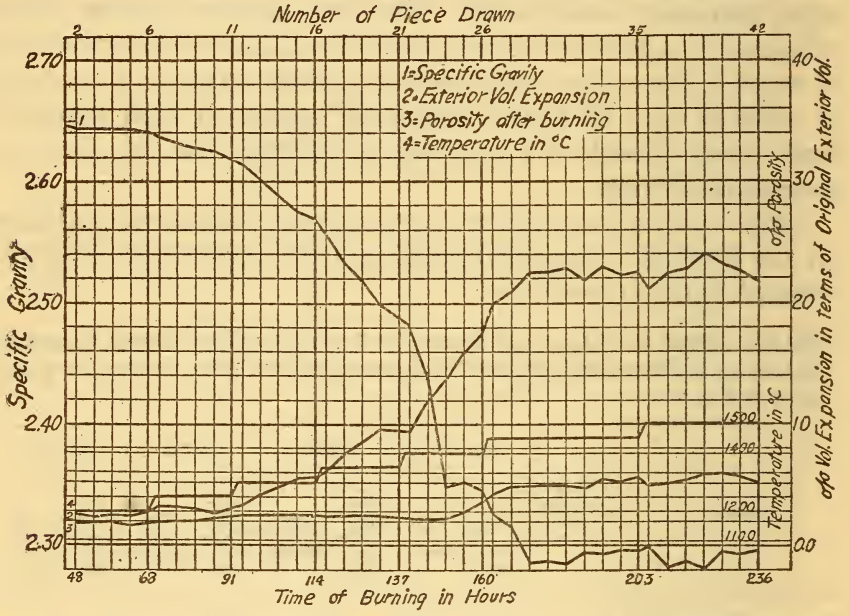

FIG. 19.—*Nature of changes in exterior volume, specific gravity, and porosity of Baraboo quartzite, due to being burned to 1500° C in 236 hours*

Points on the curves were determined by withdrawing pieces at the intervals indicated by vertical lines in the figure.

(*c*) *Series 3.*—Fig. 19 shows the exterior volume, porosity, and specific gravity curves for the 42 pieces of Baraboo quartzite. The original porosity of these pieces is not shown for it was practically zero. This burn, which also included the 42 cubes of Baraboo brick mix, more nearly approached a commercial kiln burn than the heating tests of series Nos. 1 and 2, due to the fact that it eliminated the repeated cooling down and heating up. A comparison of these two methods of burning, however, shows that the repeated burning method approximates a continuous burn fairly well. The duration of this draw trial burn was approximately the same as that of a commercial burn. It differs from a

commercial burn, however, in that a maximum temperature of 1500° C was reached.

The specific gravity curve shows that transformation did not proceed rapidly below 1350° C. This result agrees with that given in series No. 2 for Baraboo quartzite. Also in accordance with series No. 2 transformation took place rapidly above this temperature. The minimum specific gravity was reached considerably before the end of the burn and as shown is approximately 2.290. The exterior volume expansion reached its maximum, approximately 22 per cent, at the same stage at which minimum

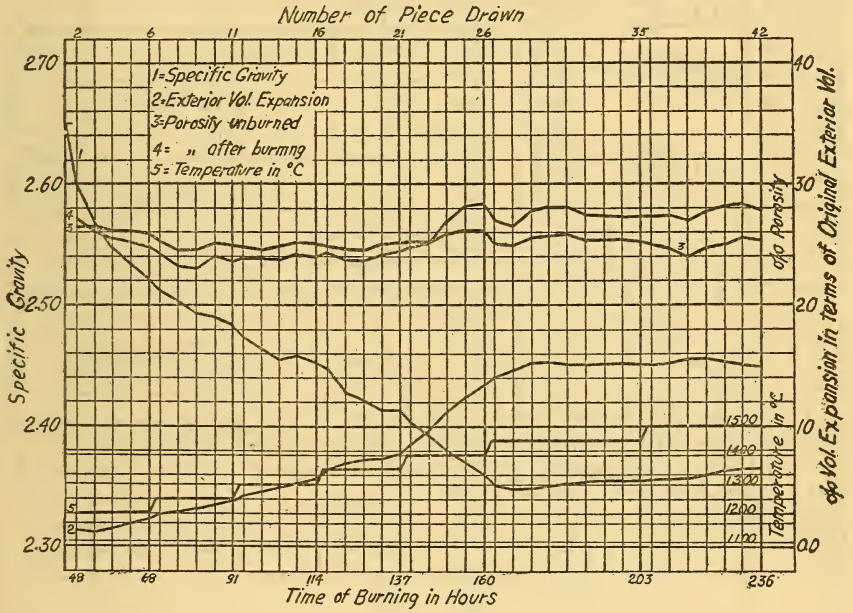

FIG. 20.—*Nature of changes in exterior volume, specific gravity, and porosity of Baraboo mix, due to being burned the same as the Baraboo quartzite of Fig. 19*

specific gravity occurred. The porosity also gradually increased up to this point, after which it remained nearly constant to the end of the burn.

The corresponding curves for the 42 Baraboo brick mix pieces are shown in Figs. 20 and 21. Fig. 21 shows the exterior and solid volume increases for the same pieces as does Fig. 20. In the case of Fig. 20, the original porosity of the pieces is also given.

These curves entirely bear out what has been said concerning the burns of series Nos. 1 and 2. In this case, however, it is plainly seen that the material has reached its maximum exterior volume expansion and its minimum specific gravity. Also in the

part of the burn between 1200 and 1350° C the porosity of the pieces was less than that of the original pieces even though considerable transformation of quartz took place during this interval, as is shown by the drop in specific gravity and increase in solid volume. In fact the expansion of the solid material in the major portion of this region was greater than or equal to the expansion of the exterior volume of the cubes. The maximum depression of the porosity was probably between 1250 and 1300° C. For the higher temperatures, however, the porosity of the burned pieces

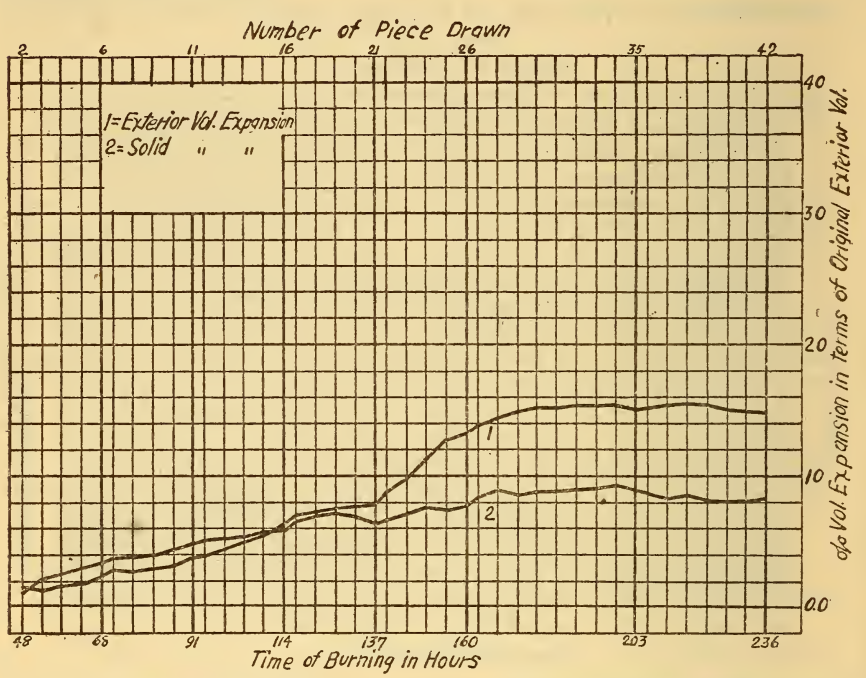

FIG. 21.—*Same materials and conditions as for Fig. 20, showing the relation between exterior and solid volume expansion*

is greater than that of the raw mix. The maximum exterior volume expansion of this brick mix is approximately 15.5 per cent and the minimum specific gravity is in the neighborhood of 2.350, as compared with 22 per cent volume expansion and 2.290 specific gravity for the quartzite pieces.

(*d*) *Series 4.*—In Table 7 the results of the single-burn tests of commercial brick are presented. As shown, these include the porosity and specific gravity of the brick as they come from the manufacturer, as well as the porosity, specific gravity, and exterior volume increase of the pieces after burning.

TABLE 7.—Changes in Porosity and Specific Gravity, and Per Cent Exterior Volume Expansion in Terms of Original Exterior Volume, of Commercial Silica Brick, Caused by Single Heatings to Various Temperatures

Number	1400° C				
	Before		After		
	Porosity	Specific gravity	Porosity	Volume expansion	Specific gravity
6	26.46	2.292	26.39	0.64	2.283
4	27.19	2.387	27.60	2.55	2.360
8	31.96	2.342	32.17	1.31	2.323
11	29.56	2.295	29.45	0.42	2.287
18	24.44	2.369	24.93	2.18	2.334
19	25.27	2.434	25.30	2.40	2.387
22	25.37	2.401	25.39	1.62	2.373
20	31.46	2.316	31.63	0.65	2.305
23	22.67	2.496	23.82	6.38	2.389
24	22.67	2.493	23.55	5.62	2.394
25	23.16	2.545	24.68	7.73	2.417
26	30.11	2.316	30.21	0.80	2.309
14	30.64	2.500	31.86	5.22	2.418
27	24.66	2.400	24.66	1.95	2.361
Average, omitting Nos. 23, 24, 25, 17, 30, and 31...	27.93	2.367	28.14	1.794	2.340

Number	1450° C				
	Before		After		
	Porosity	Specific gravity	Porosity	Volume expansion	Specific gravity
6	26.30	2.304	25.53	0.62	2.270
4	27.07	2.412	27.00	3.24	2.333
8	31.67	2.342	31.70	1.01	2.320
11	29.35	2.301	29.45	0.70	2.291
18	23.72	2.393	24.01	1.14	2.378
19	25.00	2.438	25.02	2.20	2.387
22	24.70	2.400	24.77	2.08	2.354
20	31.77	2.338	31.65	0.12	2.326
23	23.77	2.498	24.88	6.61	2.378
24	22.65	2.499	23.42	5.40	2.390
25	23.08	2.537	24.57	7.15	2.415
26	29.39	2.319	28.87	0.37	2.295
14	30.52	2.497	31.80	5.80	2.404
27	24.68	2.402	25.16	2.31	2.366
17	31.35	2.411	32.28	3.51	2.362
30	25.78	2.374	28.40	7.31	2.290
31	24.07	2.392	25.10	2.46	2.365
Average, omitting Nos. 23, 24, 25, 17, 30, and 31...	27.68	2.377	27.70	1.780	2.335

TABLE 7—Continued.

Number	1500° C				
	Before		After		
	Porosity	Specific gravity	Porosity	Volume expansion	Specific gravity
6	26.31	2.277	27.96	2.06	2.280
4	27.21	2.381	33.67	13.05	2.308
8	30.94	2.323	32.78	3.12	2.310
11	30.77	2.278	28.60	−0.13	2.281
18	23.57	2.380	25.67	4.34	2.344
19	25.17	2.417	29.50	11.10	2.334
22	24.29	2.385	28.88	8.88	2.330
20	31.11	2.322	32.06	0.73	2.334
23	23.23	2.490	30.17	17.19	2.333
24	22.67	2.475	31.20	19.70	2.322
25	22.64	2.528	31.41	22.10	2.335
26	29.76	2.298	30.45	0.37	2.306
14	30.59	2.491	38.58	20.91	2.324
27	24.56	2.378	31.55	13.05	2.353
17	30.20	2.393	36.74	13.93	2.317
30	25.67	2.353	30.74	8.80	2.317
31	25.32	2.393	29.93	9.52	2.326
Average, omitting Nos. 23, 24, 25, 17, 30, and 31	27.65	2.353	30.89	7.040	2.321

Average of determinations on original brick. Average perosity equals 27.75 per cent. Average specific gravity equals 2.366.

These properties are given for the temperatures 1400, 1450, and 1500° C. The average figures given are exclusive of Nos. 23, 24, and 25, which are materials intended for a special purpose, and Nos. 17, 30, and 31, for which the 1400° C heat is missing. The 1400° C heat was maintained for a slightly longer time than the 1450° C heat. This difference, which was first considered negligible, is noticeable both in this table and in connection with the quartzites and raw brick mixes which were also present in these burns. The near approach of the figures of the 1400° C burn to those of the 1450° C burn indicates the effect of this additional heating period.

The average figures given indicate approximately what porosity, volume, and specific gravity changes may be expected from average commercial silica brick with such heat treatments. Hence the magnitude of these changes in commercial brick for such heat treatments is a guide to the degree of burning which the bricks received in manufacture.

TABLE 8.—Changes in Porosity and Specific Gravity, Per Cent Exterior and Solid Volume Expansions in Terms of Original Exterior Volume, and of Exterior and Solid Volume Expansions in Terms of Original Solid Volume, of Quartzites and Raw Commercial Silica Brick Mixes, Caused by Single Heatings to Various Temperatures

Materials	No.	1400 C							
		Before		After					
						In terms of original exterior volume		In terms of original solid volume	
		Specific gravity	Per cent porosity	Specific gravity	Per cent porosity	Per cent exterior volume expansion	Per cent solid volume expansion	Per cent exterior volume expansion	Per cent solid volume expansion
Raw brick mixes:									
Medina	6	2.610	25.25	2.396	26.70	9.31	5.29	12.44	7.06
	4	2.614	26.37	2.409	27.46	8.11	4.63	10.99	6.28
	8	2.609	30.15	2.372	31.56	10.37	5.46	14.80	7.80
Homewood sandstone	14	2.605	28.08	2.407	30.26	9.01	4.05	12.52	5.63
Baraboo	19	2.575	25.23	2.435	26.43	5.82	2.86	7.77	3.81
Quartzites:									
Medina	6	2.615	1.27	2.516	12.61	.....	.....	.....	.....
Baraboo	22	2.650	.00	2.525	3.32	.....	.....	.....	.....
Alabama	26	2.640	.00	2.520	4.04	.....	.....	.....	.....
Homewood sandstone	14	2.628	6.54	2.487	14.68	15.16	4.80	16.21	5.14
Montana	28	2.645	.55	2.497	5.95	11.48	5.40	11.54	5.43
Soft Medina	37	.....	.....	.....	.....	.....	.....	.....	.....

Materials	No.	1450° C							
		Before		After					
						In terms of original exterior volume		In terms of original solid volume	
		Specific gravity	Per cent porosity	Specific gravity	Per cent porosity	Per cent exterior volume expansion	Per cent solid volume expansion	Per cent exterior volume expansion	Per cent solid volume expansion
Raw brick mixes:									
Medina	6	.....	.....	.....	.....	.....	.....	.....	.....
	4	2.631	25.51	2.377	27.49	11.78	6.48	15.78	8.69
	8	2.632	30.40	2.381	32.61	11.47	5.50	16.46	7.90
Homewood sandstone	14	2.617	27.75	2.427	30.32	9.47	4.01	13.11	5.55
Baraboo	19	2.615	25.47	2.444	26.95	7.25	3.73	9.73	5.00
Quartzites:									
Medina	6	2.629	1.44	2.516	10.53	14.84	4.18	15.05	4.24
Baraboo	22	2.650	.13	2.490	3.62	10.26	6.39	10.28	6.40
Alabama	26	2.642	.00	2.510	4.55	9.95	4.93	9.95	4.93
Homewood sandstone	14	2.620	6.49	2.493	14.52	14.50	4.38	15.51	4.68
Montana	28	2.641	.92	2.475	6.88	12.18	6.30	13.32	6.37
Soft Medina	37	2.615	2.25	2.573	9.32	9.26	1.34	9.47	1.36

TABLE 8—Continued.

Materials	No.	1500° C							
		Before		After					
						In terms of original exterior volume		In terms of original solid volume	
		Specific gravity	Per cent porosity	Specific gravity	Per cent porosity	Per cent exterior volume expansion	Per cent solid volume expansion	Per cent exterior volume expansion	Per cent solid volume expansion
Raw brick mixes:									
Medina	6	2.603	26.48	2.323	33.57	22.23	7.65	30.23	10.40
	4	2.640	26.03	2.333	35.85	28.50	8.28	38.50	11.20
	8	2.626	30.00	2.328	39.30	27.52	7.36	39.30	10.51
Homewood sandstone	14	2.615	28.20	2.336	35.70	22.90	6.83	31.70	9.47
Baraboo	19	2.606	24.79	2.344	31.38	19.65	6.92	26.11	9.20
Quartzites:									
Medina	6	2.635	5.60	2.303	26.76	46.70	12.00	49.40	12.70
Baraboo	22	2.650	.27	2.293	10.95	29.30	15.41	29.43	15.47
Alabama	26	2.640	.02	2.295	11.12	29.20	14.86	29.20	14.86
Homewood sandstone	14	2.630	6.52	2.326	26.64	43.40	11.66	46.40	12.50
Montana	28	2.638	1.05	2.306	22.39	45.30	13.83	45.80	13.87
Soft Medina	37	[a] 2.602	[a] 2.01	[a] 2.403	[a] 14.84	[a] 24.16	[a] 7.56	[a] 24.62	[a] 7.70

[a] In separate burn, not comparable to other figures.

In Table 8 are shown the specific gravities and porosities of raw brick mixes and quartzites before heating, their specific gravities and porosities after heating; also exterior and solid volume expansions after heating in terms of both original exterior volumes and original solid volumes.

The exterior and solid volume expansions in terms of the original exterior volumes show the behavior of any one material, and these same expansions in terms of the original solid volumes enable us to compare different materials directly. The specific gravities of the burned pieces show that up to 1450° C the brick mixes were transformed much more than were the quartzites, but that with the 1500° C heating the quartzites reached lower specific gravities than did the brick mixes. This may be due to formation of high specific gravity glass in the brick mixes. The specific gravities of the quartzites after burning show that all varieties were transformed at practically the same rate and that the 1500° C heating was sufficient to produce approximately minimum attainable specific gravity. These facts hold for the Homewood sandstone as well as for the more highly metamorphosed quartzites.

Considering the volume expansions in terms of the original solid volumes, we see that the solid volume expansions of the quartzites for the 1500° C burn is greater than that of the brick mixes. This agrees with the fact that the specific gravities of these quartzites which were burned to 1500° C is lower than that of the corresponding brick mixes when similarly burned. The exterior volume expansions of the Homewood sandstone and Medina and Montana quartzites are much greater than those of Baraboo and Alabama quartzites. This larger expansion in the case of Homewood and Medina is probably due to their structure being slightly looser than that of Alabama and Baraboo. In the case of Montana quartzite this difference may be due to its being much finer grained than any other materials tested. These phenomena apparently also hold for the brick mixes; thus, brick made from Baraboo or Alabama quartzites would not be expected to increase as much in porosity during burning as would those made from the other varieties shown in this test. This agrees with our other data on this subject.

(*e*) *Series 5.*—In connection with added ingredients the results of the first test are given in Table 9. These figures show that a much greater transformation took place in the case where lime plus shale was added than where shale alone was added. It is also apparent that 2 per cent of shale in conjunction with lime produced considerably more transformation than did 1 per cent of Fe_2O_3 in conjunction with lime.

TABLE 9.—Effects of Added Ingredients, Judged by the Percentage Exterior Volume Expansion of Various Mixtures, Which Were Heated Twice to 1400° C in Terms of Original Exterior Volume

Material	No.	Per cent volume expansion, first burn to 1400° C	Per cent total volume expansion after second burn to 1400° C	Per cent expansion caused by second burn
Medina Quartzite plus 2 per cent CaO plus 1 per cent Fe_2O_3	1	10.82	11.82	1.00
Medina Quartzite plus 3 per cent Metropolitan shale	2	4.48	5.80	1.32
Medina Quartzite plus 2 per cent CaO plus 2 per cent Metropolitan shale	3	11.16	13.45	2.29

In Table 10 are shown the results of the second test. In general it may be said that the ingredients added in this test, other than lime, did not appear to alter the rate of transformation or porosity change greatly at the temperature used (1450° C). Lime did, however, greatly increase the rate of transformation and larger percentages apparently increased the rate still more. This speed-

ing up of the rate of transformation apparently causes greater increase in porosity than does a corresponding amount of transformation at a lower rate.

TABLE 10.—Effects of Added Ingredients, Judged by Specific Gravity, Porosity, and Exterior Volume Changes of Various Mixtures Which Were Heated to 1450° C

Materials	No.	Before heating		After heating		Per cent exterior volume expansion
		Specific gravity	Per cent porosity	Specific gravity	Per cent porosity	
Medina plus 2 per cent CaO	1	2.615	38.4	2.405	40.20	9.60
Medina	2	2.615	2.2	2.573	9.32	9.25
Medina plus 3 per cent Metropolitan shale	3	2.530	37.0	2.520	43.50	6.30
Medina plus 2 per cent Tennessee ball clay	4	2.610	40.4	2.568	42.70	4.32
Medina plus 2 per cent CaO plus 2 per cent Metropolitan shale	5	2.630	36.7	2.389	38.80	11.80
Medina plus 2 per cent CaO plus 2 per cent Tennessee ball clay	6	2.630	38.8	2.427	41.00	9.77
Medina plus 2 per cent CaO plus 1 per cent Al_2O_3	7	2.630	39.1	2.468	41.70	9.10
Medina plus 2 per cent CaO plus 2 per cent Al_2O_3	8	2.640	37.6	2.496	39.20	5.94
Medina plus 0.5 per cent CaO plus 1.5 per cent CaF_2	9	2.560	35.3	2.413	40.20	9.49
Medina plus 2.5 per cent CaO	10	2.615	30.9	2.389	33.70	11.53
Glass sand plus 2 per cent CaO	11	2.620	33.2	2.562	36.02	4.53

Table 11 shows the results of the third test (four-day burn). The coarse grind which the material received does not appear to have materially altered the properties of the burned pieces. The effects of pressure were readily discernible, however, as characteristic numerous fine cracks over the surface of all the pieces after burning. In this test all the pieces approximately reached minimum specific gravity during burning. However, a decided effect of the sodium chloride is shown by the exceptionally great increase in porosity on burning of the mix containing it.

TABLE 11.—Effects of Added Ingredients and Molding of Brick Under Pressure, Judged by Specific Gravity, Porosity, and Exterior Volume Changes of Various Mixtures, Which Were Heated to 1450° C in Four Days

Materials	No.	Before heating		After heating		
		Specific gravity	Per cent porosity	Specific gravity	Per cent porosity	Per cent exterior volume expansion
Medina plus 2 per cent CaO aepressed 5000 pounds per square inch	1	2.630	28.10	2.321	35.80	24.10
Medina plus 2 per cent CaO plus 2 per cent Metropolitan shale 5000 pounds per square inch	2	2.630	27.52	2.321	34.90	24.23
Medina plus 2 per cent CaO plus 1.4 per cent NaCl 5000 pounds per square inch	3	2.640	27.74	2.312	41.30	.....

In considering all the tests that have to do with added ingredients, the indications are that rate of transformation is increased by lime or sodium chloride, very slightly increased by impure ferruginous clay when used in conjunction with lime, and is practically not affected by pure plastic clay or the other ingredients which were tested. In this connection it must be remembered that the above tests are special cases and that in nature the foreign ingredients would in all probability be much more intimately associated with the quartz grains and thus cause somewhat different results.

In connection with the tests of calcined material a microscopic examination of the quartzites after the second calcination revealed the fact that they were largely converted to cristobalite, but that small interior portions still remained as unaltered quartz. In the first of these tests the Medina and loose-grained Medina mixes crumbled and fell apart during burning, while the Baraboo mix held together, but had no appreciable strength. In the second test, in which the test pieces were molded under a pressure of 5000 pounds per square inch, the results were the same. One piece of Baraboo mix was found to be strong enough after burning to allow of its volume being measured. This showed an exterior volume increase of approximately 3 per cent.

The above data indicate that strong brick can not be made from strongly calcined material, at least when the duration of burning of the brick is only a few days. However, the permanent exterior volume increase of such material is small.

(*f*) *Series 6.*—The materials of the first burn of the test of soft Medina quartzite contained 2 per cent of lime. It was found that 91.6 per cent of the 8-mesh quartzite went through a 20-mesh screen before being mixed with the lime. Apparently the 8-mesh material yielded a considerably more durable brick than the 10-mesh grind. However, all brick of this burn were too friable to be termed first class.

The results of the later tests of this series are shown in Table 12. As is shown under burn No. 3, the 1 per cent of lime plus 1½ per cent of Tennessee ball clay bricks expanded more than did the ones where lime alone was used, and the bricks which contained 2.5 per cent of Tennessee ball clay alone expanded less than the bricks that contained lime.

TABLE 12.—Effects of Added Ingredients, Judged by Linear Expansion of Various Mixtures, Which Were Given Heat Treatments of Varying Duration and Temperature

No.	Materials	Burn No.	Piece No.	Through mesh	Cone No.	Maximum temperature	Per cent linear expansion	Total per cent linear expansion
37	Medina previously calcined to 1300° C 2 per cent CaO.	2	2	8	17	1485 flash	3.15	.....
37	Medina plus 2.5 per cent CaO	3	3	8	16+	1485 flash	3.77	.....
37	Medina plus 3.0 per cent CaO		4	8			3.72	.....
37	Medina plus 1.0 per cent CaO plus 1.5 per cent Tennessee ball clay.		5	8			4.07	.....
37	Medina plus 2.5 per cent Tennessee ball clay.		6	8			3.69	.....
		4	2	.....	.....	1450° C for 5 hours	1.70	4.85
			3				1.85	5.62
			4				2.11	5.83
			6				2.28	5.97
37	Medina plus 2.5 per cent CaO	5	7	8	20	1470° C for 3 hours	5.40	.....
37	Medina plus 2.5 per cent CaO		8	20			6.33	.....
39	Hard quartzite plus 2.5 per cent CaO.		9	8			7.32	.....

In the fourth burn mixes Nos. 3, 4, and 6 (Table 12) approached a common percentage expansion, while the expansion of mix No. 2 was considerably less. This seems to indicate that in this burn the materials reached approximately the limit of expansion, and that this limit was decreased in the case of No. 2 by the amount of transformation that had taken place in the calcining of the quartzite. Burn No. 5 shows a decided difference in expansion between the 8-mesh and 20-mesh material. The 20-mesh material after burning had no appreciable strength. This lot of brick, however, were made up somewhat drier than the 8-mesh brick. This may have had something to do with their friableness.

The hard rock mix No. 9 shows a greater expansion than the soft rock tested. It yielded a brick which compared favorably with commercial brick as far as ring and spalling tendency are concerned.

In the several tests above with loose-grained Medina quartzite, the exterior volume expansion did not exceed 6 per cent except with the most friable brick. Although several combinations gave fairly satisfactory brick, the most satisfactory probably being 8-mesh quartzite plus 2.0 per cent of lime. None of them had the best ring and all showed a decided tendency to spall.

(*g*) *Series 7.*—Table 13 gives the data of the burn in a commercial kiln. Nos. 1, 2, and 3 are the same mixes as Nos. 1, 2, and 3 of Table 11, and received the same pressure in molding. No. 4 was a mix from some of the commercial Medina which had been calcined twice to 1500° C. This piece was subjected to 5000 pounds per square inch when molded. No. 5 was a mix made from the clacined Baraboo quartzite.

TABLE 13.—Effects of Added Ingredients, Molding of Brick Under Pressure, and the use of Calcined Quartzites, Judged by Specific Gravity, Porosity, and Exterior Volume Changes of Various Mixtures, Which Were Heated to Cone 16+ in 14 Days in a Commercial Kiln

Materials	No.	Before heating		After heating		
		Specific gravity	Per cent porosity	Specific gravity	Per cent porosity	Per cent exterior volume expansion
Medina plus 2 per cent CaO, repressed 5000 pounds per square inch	1	2.642	29.27	2.362	31.05	14.83
Medina plus 2 per cent CaO plus 2 per cent Metropolitan shale, 5000 pounds per square inch	2	2.640	27.82	2.347	31.10	16.20
Medina plus 2 per cent CaO plus 1.4 per cent NaCl, 5000 pounds per square inch	3	2.640	28.03	2.313	39.02	36.20
Medina strongly calcined plus 2 per cent CaO	4	2.310	31.64	2.310	35.00	2.48
Baraboo strongly calcined plus 2 per cent CaO	5	2.310	29.25	2.283	30.30	1.28

The specific gravities of 1, 2, and 3 after burning show that the material containing the shale and lime were transformed somewhat more than that containing lime alone, and that the material which contained sodium chloride and lime was transformed considerably more than either of the other two. Its porosity increased to practically double that of either of the others. Its structure was so friable that it barely held together while being measured. It could be crumbled readily between the fingers, whereas the other two were moderately strong.

Comparing the porosities of 1, 2, and 3 with 1, 2, and 3 of Table 11 we see that apparently the former had appreciably lower porosities than the latter, also inspection showed that pieces Nos. 1 and 2 of the latter were much more friable than those burned in the commercial kiln. The fine surface cracks, apparent in the repressed material of the short-time burn, were also present in the commercially burned pieces. The pieces present in this commercial burn, which were made from highly calcined quartzites, showed very little volume increase, and had practically no strength when

burned, which facts bear out what was found to be true for a short-time burn.

(*h*) *Series 8.*—Table 14 shows the changes in specific gravity and porosity brought about in the underburned, medium burned, and well-burned bricks, made from Medina quartzite by rapid reheating.

TABLE 14.—Effects of Rapid Reheating to 1480° C, on Underburned, Medium Burned, and Well-Burned Brick, Made of Medina Quartzite

Materials from one kiln [a]	No.	Before heating		After heating to 1480° C		
		Specific gravity	Per cent porosity	Specific gravity	Per cent porosity	Condition of brick
Medina brick No. 6:						
Soft burn	1	2.50	30.1	2.32	39.6	Cracked.
Medium burn	2	2.46	27.9	2.32	36.4	Do.
Hard burn	3	2.32	33.5	2.33	34.1	Fairly sound.

[a] Effects judged by changes in specific gravity, porosity, and amount of cracking.

In the first place this table indicates what a wide range in the degree of burning may occur in a single kiln of bricks. It also indicates that in a moderately burned brick the porosity is lower than in either underburned or well-burned brick. The high porosity and great decrease in specific gravity of brick No. 1 after heating shows what rapid heating does to underburned brick; similarly, No. 2 shows the undesirable effect produced in a medium burned brick; and No. 3 shows what a relatively small porosity change is occasioned in the case of a well-burned brick. This evidence is strongly in favor of a brick in which the silica has been largely transformed from quartz to the other crystal forms.

2. PROPERTIES OF FINISHED BRICK AND DATA ON MANUFACTURE

(*a*) *Data on Commercial Bricks.*—When it was once determined that the specific gravity of a silica brick was a definite measure of the degree to which the brick had been burned, it became desirable to know what the degree of accuracy of such measurement is, whether all varieties of material would fall within these limits, and what the average specific gravity of the leading varieties of brick is.

The specific gravity of raw quartzite is approximately 2.650 and the specific gravities of artificial cristobalite and tridymite are approximately 2.333 and 2.270, respectively. Hence the

specific gravities of brick should lie between these two extreme points. The figures of Table 15 represent the specific gravities of 12 pieces of raw American quartzites.

TABLE 15.—Check Specific Gravity, Determinations made on Quartzites by the Wet, Dry, and Suspended Weight Method, to Determine the Degree of Agreement Between Separate Determinations by this Method, Using a Vacuum Equal to 24 Inches of Mercury, to Insure Saturation

	Group 1	Group 2
Close-grained material	2. 650	2. 650
	2. 650	2. 650
Mean	2. 650	2. 650
Open porous material	2. 620	2. 604
	2. 610	2. 600
Mean	2. 615	2. 602
Close-grained material	2. 646	2. 642
	2. 638	2. 638
Mean	2. 642	2. 640

Two of these varieties are nearly nonporous and the third is an open-porous variety. Each set of two determinations for a variety were made on the same day, but the different groups for a variety were determined at different times. The degree of agreement of the determinations for any one variety can be seen from the figures. Considerably over a thousand such determinations have been made with quartzites, bricks, etc., and the degree of agreement of the determinations has been quite generally as close as the ones presented. A series of specific gravity determinations were also made on these quartzites by means of a picnometer, and were found to check the above results as closely as the individual picnometer readings for a variety checked each other.

To determine whether all varieties of material would be likely to fall within these limits a number of unusual varieties of material were tested. The results of these tests are shown in Table 16.

TABLE 16.—Properties of Some Unusual Silica Brick Materials

Materials	No.	Specific gravity	Per cent porosity
Medina quartzite brick in kiln with Baraboo draw trial burn 1 day at 1500° C, 4 days at 1450° C+	6	2. 321	29. 45
Medina quartzite brick heated 40 times on bag wall of test kiln to 1450° C	6	2. 275	31. 85
Brick made from Baraboo quartzite which received 9 years' service in Koppers by-product coke oven	18	2. 314	19. 94
German brick, Stella Werke* * *	33	2. 372	20. 07
Homewood sandstone	14	2. 626	6. 52
Brick made from Homewood sandstone	15	2. 448	28. 95
California quartzite plus California River sand plus 2 per cent of CaO; 2 heatings in commercial clay brick kiln	34	2. 437	30. 02
Raw Indiana chert rock	29	2. 585	1. 83
Brick made from Indiana chert	29	2. 273	25. 64
Alabama mica quartz schist	35	2. 690	4. 52
Brick made from mica quartz schist	35	2. 616	34. 07
Brick made from slightly impure silica sand plus 8 per cent CaO $(OH)_2$	36	2. 527	30. 40
Brick made from Medina quartzite that received 10 commercial burns to cone 14	6	2. 271	27. 25
Brick made from West Virginia quartzite	38	2,270	28.40

From this table we see that the specific gravities of products which have seen service at high temperatures may be considerably above the theoretical limit, and even appreciably above the specific gravities of some unused bricks made from Medina quartzite. Likewise, the German-made brick, the Homewood sandstone and the brick made from it, as well as the brick made from a mixture of quartz and California river sand are well within the limits. The Indiana raw chert rock is of interest because its specific gravity is considerably lower than that of quartz. Likewise, the specific gravity of the resultant brick is near the lower limit. These matters concerning chert, however, are entirely in accordance with the work of Mellor, given above, which shows that the original specific gravity of this kind of material is approximately 2.61 before heating, and reaches 2.22 as a final limit when repeatedly heated to cone 10, and also that the rate of transformation of this material is much more rapid than that of quartz, when heated to the same temperature. This view with respect to the rate of transformation of chert is also borne out by the work of Khoetsky and Riekie and Endell, also cited above. The mica quartz schist and the brick made from it are of interest in that they both have a specific gravity above 2.65 on the opposite side of the permissible range from chert. This is to be expected, however, from the fact that mica (probably muscovite in this case) has a specific gravity somewhere between 2.70 and 3.10.

This particular material is outside the range of usefulness for silica brick, however, as its softening temperature is too low.

Some question has been raised as to the necessity of a vacuum for four hours in saturating pieces for specific gravity and porosity determinations. Hence the tests shown in Table 17 have been carried out by obtaining saturation by boiling in water for four hours without subjecting to a vacuum.

TABLE 17.—Check Specific Gravity Determinations Made on Burned Silica Bricks, by the Wet, Dry, and Suspended Weight Method to Determine the Degree of Agreement Between Separate Determinations, when Saturation is Obtained by Boiling in Water for Four Hours

Materials	Approximate size and weight of piece							
	2-inch cube, 260 g, 4 hours in boiling water		1.25-inch cube, 65 g, 3 hours in boiling water		0.75-inch cube, 18 g, 2 hours in boiling water		0.5-inch cube, 10 g, 1 hour in boiling water	
	Specific gravity	Per cent porosity	Specific gravity	Per cent porosity	Specific gravity	Per cent porosity	Specific gravity	Per cent porosity
No. 6. Brick made from Medina quartzite, soft burn	2.47	29.0	2.48	29.2	2.51	28.8	2.49	30.4
	2.49	28.4	2.49	28.3	2.49	29.2	2.48	29.1
No. 6. Brick made from Medina quartzite, well burned, good ring	2.31	28.9	2.33	29.0	2.34	28.7	2.32	28.0
	2.30	28.7	2.33	29.2	2.33	26.3	2.32	27.0
No. 28. Brick made from Colorado quartzite	2.43	26.3	2.43	27.0	2.44	27.7	2.41	26.8
	2.40	26.1	2.43	26.9	2.44	27.5	2.42	26.8
No. 29. Indiana chert brick	2.27	24.8	2.29	24.1	2.30	24.2	2.28	24.0
	2.27	24.1	2.28	23.0	2.30	23.8	2.28	22.5

For the range of materials tested the degree of agreement appears satisfactory for our purpose. As far as tested these results also appear to check those obtained by use of the vacuum. However, wherever practicable it is safest to use the vacuum.

In making specific gravity and porosity tests on a commercial brick it is well to take two pieces for test, from different parts of the brick. If they do not check, it is well to repeat the test to determine whether the difference is due to error or to actual variations in the brick. It is advisable always to have the pieces large enough so that the presence of large grains will not cause errors in porosity.

Considering Table 18, we see that the average specific gravity of all varieties of brick tested is 2.384, and the average porosity is 26.35 per cent. The average specific gravity shown by commercial brick made from Medina quartzite is 2.357, and the lowest porosity shown is 2.291. The porosities of Medina brick range from 23.10 to 31.52. The average specific gravity of brick made from

Baraboo quartzite is 2.399 and the lowest specific gravity of a single-burn brick is 2.381. The porosities of Baraboo brick range from 23.45 to 25.15.

TABLE 18.—Specific Gravities and Porosities of some of the Chief Brands of Commercial Silica Brick Manufactured in the United States, including a list of the Quartzites From Which They Were Made

Quartzite from which brick were made	No.	Specific gravity	Per cent porosity
Medina (Tuscarora)	1	2.375	27.15
	2	2.296	25.84
	3	2.424	28.52
	4	2.393	27.16
	5	2.340	22.80
	6	2.291	26.35
	7	2.375	28.35
	8	2.336	31.52
	9	2.321	25.97
	10	2.358	26.10
	11	2.291	29.56
	12	2.480	23.10
	13	2.340	29.88
Average		2.357	27.07
Homewood sandstone	14	2.496	30.58
	15	2.448	28.95
	16	2.517	26.55
Average		2.470	28.69
Oneida	17	2.274	25.66
Baraboo (No. 20 made from lightly calcined quartzite)	18	2.381	23.91
	19	2.430	25.15
	20	2.325	31.41
	21	2.390	23.45
	22	2.395	24.79
Average		2.399	24.30
Quadrant formation (Montana)	23	2.495	23.22
	24	2.490	22.66
	25	2.537	22.96
Alabama, probably Weisner Formation	26	2.311	29.75
Dakota and Comanchian (Colorado)	27	2.393	24.63
	28	2.387	24.51
Indiana chert (Mitchell formation)	29	2.273	25.64
Saint Louis, Mo., district: Crypto crystalline silica	30	2.363	25.73
Saint Louis, Mo., district: Chalcedony bonded sand stone	31	2.393	24.70
Eastern Pennsylvania (probably Chicke's)	32	2.335	26.85
Grand average		2.384	26.34

With other varieties of material the brands of brick are too few to be of value for general conclusions. It is of interest to note that in the case of the chert brick, the specific gravity is 2.273 and

the porosity 25.64 per cent. The data concerning time and temperature of burning during manufacture of the bricks represented in this table are so incomplete and inaccurate that their presentation would only cause confusion. However, the data at hand indicate that bricks made from quartzite and having a specific gravity below the average (2.384) have been burned from cone 18 to 20.

The above figures can be considered representative of silica refractories, as they are manufactured in the United States at the present time.

(*b*) *Analyses and Softening Temperatures.*—A typical analysis [24] of brick made from Medina quartzite is as follows:

	Per cent
SiO_2	96.25
Al_2O_3	.89
Fe_2O_3	.79
CaO	1.80
MgO	.14
Alkali	.39
	100.26

Brick having an analysis such as this should have a softening temperature somewhat above that of Orton cone 32.

Brick No. 15, of Table 16, which was made of Homewood sandstone, has a softening temperature above that of cone 32, and brick made from Baraboo quartzite usually have softening temperatures above that of cone 31.

A German silica block (Stella Werke * * *) has an analysis as follows:

	Per cent
SiO_2	94.20
Al_2O_3	1.60
Fe_2O_3	1.62
CaO	2.24
MgO	.09
	99.75

Its softening temperature was found to be somewhat above that of Orton cone 30.

[24] K. Seaver, Manufacture and Tests of Silica Brick for the By-Product Coke Oven, Am. Inst. Min. Eng. Transactions, 53, pp. 125–139; 1916.

Analyses of the raw Indiana chert and of brick made from it are as follows:

	Chert	Brick		Chert	Brick
	Per cent	Per cent		Per cent	Per cent
SiO_2	96. 67	95. 60	K_2O	0. 16	0. 16
Fe_2O_3	. 48	. 61	Na_2O	. 05	. 06
Al_2O_3	1. 59	1. 80	Ignition	1. 26	. 12
CaO	. 07	2. 01			
MgO	Nil	Nil		100. 28	100. 36

The softening temperature of the brick is somewhat above that of cone 30.

The analysis of a brick (No. 36, Table 16) made from slightly impure silica sand and 8 per cent of hydrated lime is as follows:

	Per cent
SiO_2	90. 44
Al_2O_3	. 94
Fe_2O_3	1. 10
CaO	6. 50
MgO	. 81
Na_2O	. 11
K_2O	. 16
Ignition	. 12
	100. 18

The softening temperature of this brick is the same as that of Orton cone 31.

Brick made from the Quadrant Formation (Montana) quartzite had a softening temperature somewhat above that of cone 30, and the Colorado quartzites also yielded brick with approximately this softening temperature. Tests of other lots of brick made from these Montana and Colorado materials have shown softening temperatures somewhat higher than these, but in each case they were below that of cone 32.

With reference to the effect of high iron content, it is interesting to note that material No. 10, Table 18, contains 1.60 per cent of Fe_2O_3 and its softening temperature is above that of cone 32. This iron is present as grains of iron oxide. This is in accordance with the work of those [25] who found 15 to 17 per cent of magnetite in the darkened glassy surface portion of brick that had become "seasoned" through use in a steel furnace. The seasoning process apparently made them less subject to injury from changes in temperature, etc.

[25] Silica as a Refractory Material, Cosmo Johns, Trans. of the Faraday Society, 12, parts 1, 2, and 3.

Material No. 18, Table 16, which was made from Baraboo quartzite, received nine years' service as part of the wall of a Koppers by-product coke oven. It was between the coking chamber and a heating flue, 20 feet from the pusher end and 6 inches above the floor of the coking chamber. When the oven was in operation, the average temperature on the coking side of the brick was 1110° C (2000° F), the average on the heating flue side was 1330° C (2400° F) and the maximum on the heating flue side was 1550° C (2800° F). A sample of the brick that had been in contact with the coke possessed a softening temperature above that of cone 32, while samples from the interior of the brick and the side which was exposed to the heating flue gasses had a softening temperature somewhat above that of cone 31. (See other properties of this material below under (*d*).)

The above analyses and softening temperatures show in a limited way what a variety of substances come within the range of chemical composition of materials suitable for silicia refractories.

It is apparent from these analyses and softening temperatures that appreciable quantities of iron and alumina (up to 1.60 per cent of each) may not greatly lower the softening temperature of a brick. From these analyses it is also apparent that in all cases the total alkalies present must be fairly low (not usually exceeding 0.40 per cent). In all tests where much larger quantities have been present the softening temperature has been found to decrease rapidly with increase of alkali.

(*c*) *Load tests.*—In running load tests on silica brick, it was soon learned that if heated to 700° C at the same rate as is usually followed with clay brick (270° C in 20 minutes, 520° C in 40 minutes, and 690° C in 60 minutes), the specimen invariably spalled. Hence, a rate of 50° C in 15 minutes to 700° C was tried. No spalling occurred. This rate of heating has since been used with many varieties of silica brick and in no case has spalling of the brick occurred. Increasing the rate to 100° C in 15 minutes above 500° C seldom if ever caused spalling.

These tests frequently repeated have lead to the conclusion that practically all spalling of silica brick occurs between atmospheric temperature and 500° C.

A brick of the same variety as No. 27, Table 18, was subjected to a load test of 25 pounds per square inch and a maximum temperature of 1400° C maintained for one and one-half hours, and a record was kept of its change in length by means of two catheto-

meters, one sighted on the base of the brick and one on its top. The results are tabulated in Table 19.

TABLE 19.—Expansion of Commercial Silica Brick No. 27, While Being Load Tested to 1400° C, Under a Pressure of 25 Pounds Per Square Inch

Time in hours	Temperature in degrees centigrade	Per cent linear expansion
0. 00	19	0. 0
1. 00	200	. 0
2. 50	500	. 340
3. 00	650	. 363
4 50	1200	. 408
4. 75	1250	. 408
5. 00	1300	. 431
5. 25	1330	. 431
5. 50	1360	. 657
6. 00	1400	. 657
6. 50	1400	. 657
7. 00	1400	. 703
7. 50	1400	. 840
Measured cold after test		. 158
Total expansion		. 840
Permanent expansion		. 158
Difference compared to 0.657 at 1360° C		. 682

These figures show that from atmospheric temperature to 300° C a decided expansion took place, and that this maximum point in the curve did not flatten out again until a temperature of approximately 500° C was reached. In other words, approximately one and one-half hours were consumed before this expansion was complete. Above this point there was a small gradual change in length until the maximum temperature (1400° C) was reached, at which temperature a rather decided gradual expansion in length took place. It is also seen that the amount of this latter expansion is practically equal to the amount of permanent expansion of the brick. The figures in this table indicate how difficult and inaccurate it would be to endeavor to express the volume changes of a silica brick caused by heating, in terms of a single thermal expansion factor.

Another of these same brick was subjected to a load test of 25 pounds per square inch and 1500° C as the maximum temperature, which was held for one and one-half hours. In this case the permanent expansion in length was 2.6 per cent, and the load carrying lever arm of the test furnace showed that a decided increase

took place above 1400° C. Other tests of this nature on several varieties of brick have yielded results which are in accord with the above data.

The low temperature volume changes are apparently those caused by the low temperature tridymite inversions (below 200° C), which are accompanied by slight volume changes, and by the Alpha-Beta cristobalite inversion at from 220 to 275° C, which is accompanied by a considerable volume change. The expansion that took place just above 300° C is evidently due to the lag in the penetration of the heat to all parts of the specimen and to the time required for the inversions to take place. Since the percentage of tridymite in most commercial silica brick is quite small, the effect due to the tridymite inversions is apparently negligible. It is also apparent that the slow expansion at high temperatures is due to actual transformation of quartz. In the case of bricks which have been greatly underburned in manufacture, the volume change accompanying the Alpha-Beta quartz inversion at 575° C is apt to cause a slight increase in length at this temperature. These conclusions are in accordance with the results of Fulweiler's work presented above.

(*d*) *Micro Studies.*[26]—Several pieces of the Baraboo draw trial burn (series 3) were examined under the microscope; these included both the quartzite alone and the brick mix. It was apparent from these that cristobalite formed first on the surface of the grains and along cracks through them. The glass present in these pieces had an index of refraction considerably higher than the silica minerals.

This indicates that in the individual grains the transformation of quartz to cristobalite proceeds from the surface to the center, and since high index of refraction indicates high specific gravity in the case of such glasses, it is probable that the presence of much such high index glass may at times tend to cause slightly higher specific gravity than would otherwise be the case.

Material No. 6 (second position) of Table 16, a view of which is shown in Fig. 22, was heated approximately 40 times to an average temperature of 1450° C for short periods. The ground mass is practically all tridymite. The larger particles are either grayish in color or have white centers in a gray outer portion. The white central portions were found to be fine grained and to consist of cristobalite, while the gray material was found to be coarser

[26] A good deal of this information was privately communicated by Dr. C. N. Fenner of the Geophysical Laboratory, Washington, D. C. For other data the writer is indebted to Mr. H. Insley.

grained and to consist of tridymite and a glass of high index of refraction. As shown in Fig. 22, the white spots surrounded by darker bands are the larger particles which show the cristobalite centers surrounded by tridymite and glass.

Consideration of the above studies indicates that the quartz is converted to cristobalite from the surface to the center of the individual grains; that in brick which are mostly cristobalite the ground mass is the first to change from cristobalite to tridymite, and that the larger particles pass progressively from cristobalite to tridymite from the outside to the interior. The latter test shows that even after such an extended heat treatment some cristobalite still remains.

A material from Colorado which is used in small percentages in manufacturing silica brick was found to be a quartz conglomerate bonded with chalcedony, and a material from Missouri (No. 31), which is used alone in the manufacture of silica brick, was found to consist of a medium grained silica sandstone, bonded with a mixture of chalcedony and fine quartz crystals, which crystals are not of the same orientation as the original sand grains.

The Indiana chert was examined and like other cherts it was found to be cryptocrystalline. Scattered through it at wide intervals small groups of quartz crystals were found, which are plainly visible to the naked eye.

The brick made from this chert, and which received the usual burn in a commercial kiln, was found to consist of quartz plus silicate 5 per cent, cristobalite 12 per cent, and tridymite 83 per cent. This brick is of special interest on account of the very large percentage of tridymite which is present in it as a result of a single commercial burn. A comparison of this with the microanalyses of brick made from quartzites shows this plainly.

Material 30, Table 7, is composed of cryptocrystalline silica from southern Illinois and is of interest in that, as far as studied, its behavior is similar to that of chert (No. 29) discussed above, except that much of the southern Illinois rock is apparently composed of grains, practically all of which will pass through a 330-mesh screen, and which are but slightly banded together, while the Indiana chert is much harder and approaches a massive structure.

The German brick (Stella Werke * * *), material No. 33 of Table 16, was examined and found to contain quartz + silicates 20 per cent, cristobalite 78 per cent, and tridymite 2 per cent. This microanalysis in conjunction with the low porosity and high

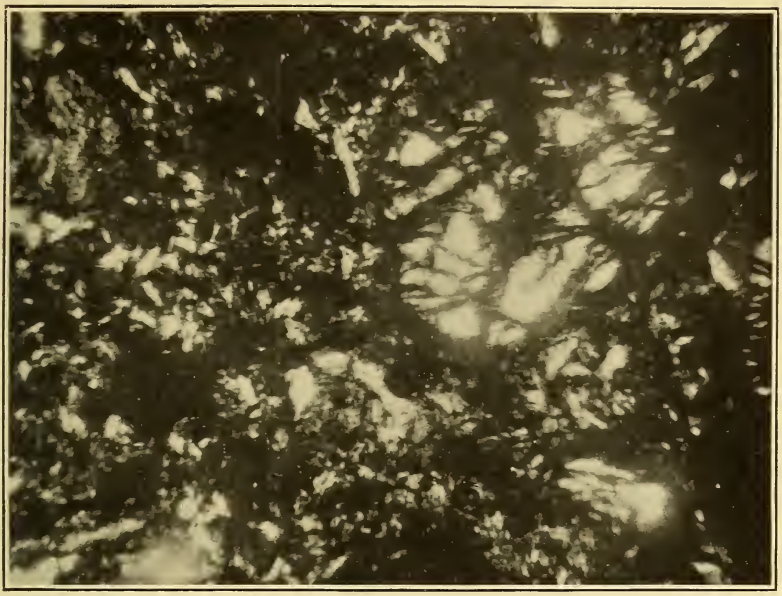

FIG. 23.—*Commercial brick, Medina quartzite, taken with crossed nicols (magnification 140 diameters), and showing large white patches are quartz grains that have been converted to cristobalite along cracks in the grains; white wedge-shaped particles in groundmass are tridymite twins*

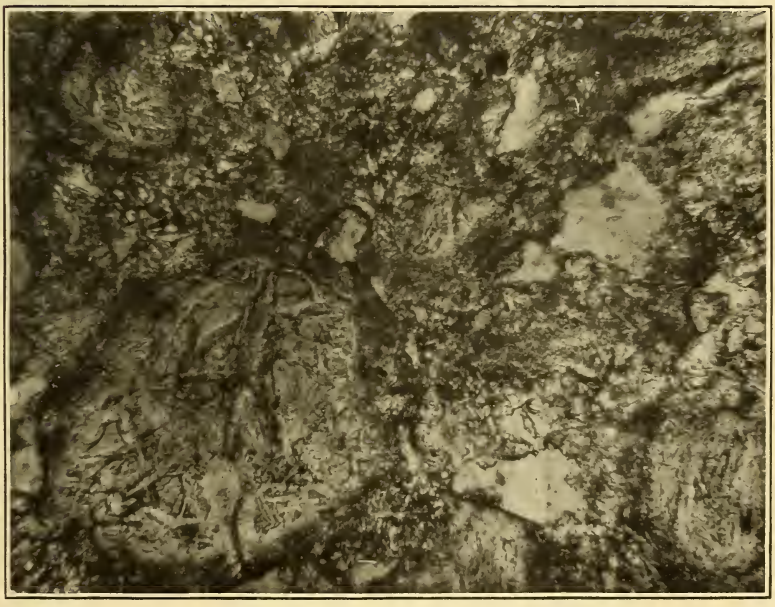

FIG. 24.—*Commercial brick, Medina quartzite, ordinary light (magnification 140 diameters), showing large quartz grains scattered through groundmass of finer material; quartz grains transformed to cristobalite along cracks*

FIG. 25.—*Commercial brick, Baraboo quartzite, crossed nicols (magnification 125 diameters). Large quartzite grains seamed by cristobalite show up well, also a few tridymite twins*

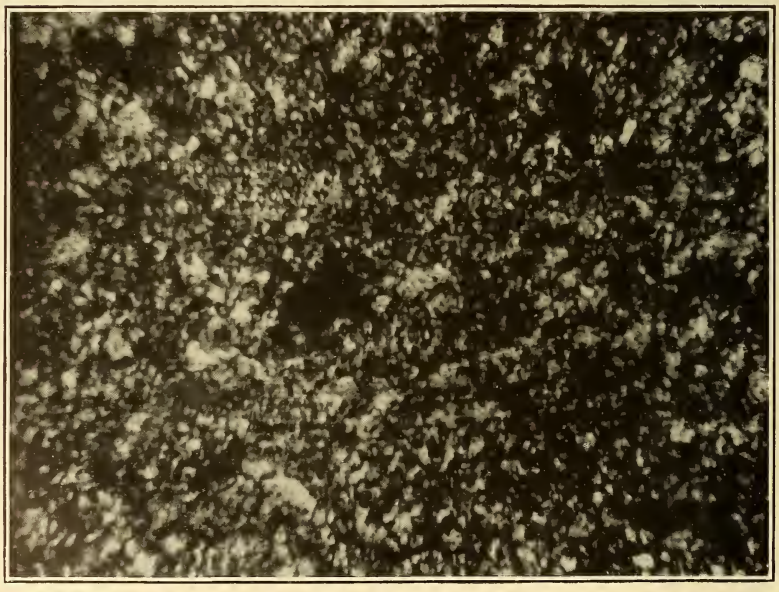

FIG. 26.—*Raw Indiana chert rock, crossed nicols (magnification 140 diameters), showing granular structure*

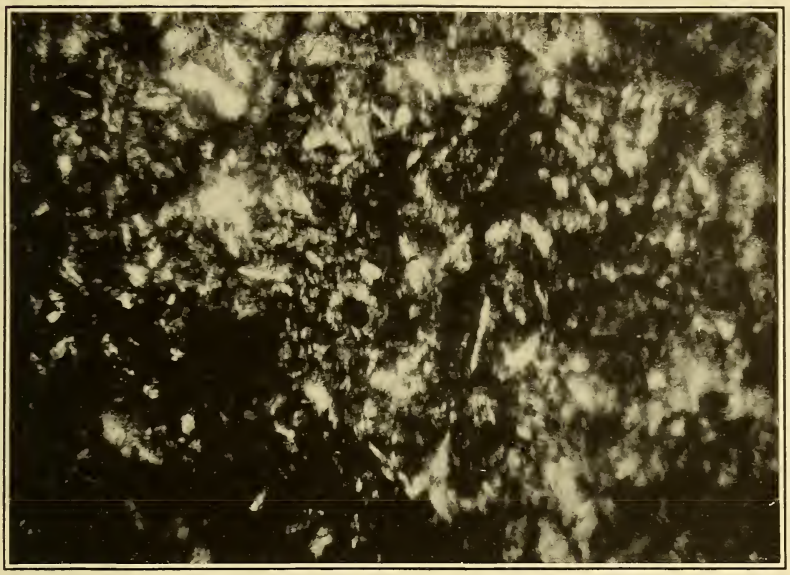

FIG. 27.—*Commercial brick, Indiana chert, crossed nicols (magnification 140 diameters), mostly tridymite, showing great quantities of tridymite twins*

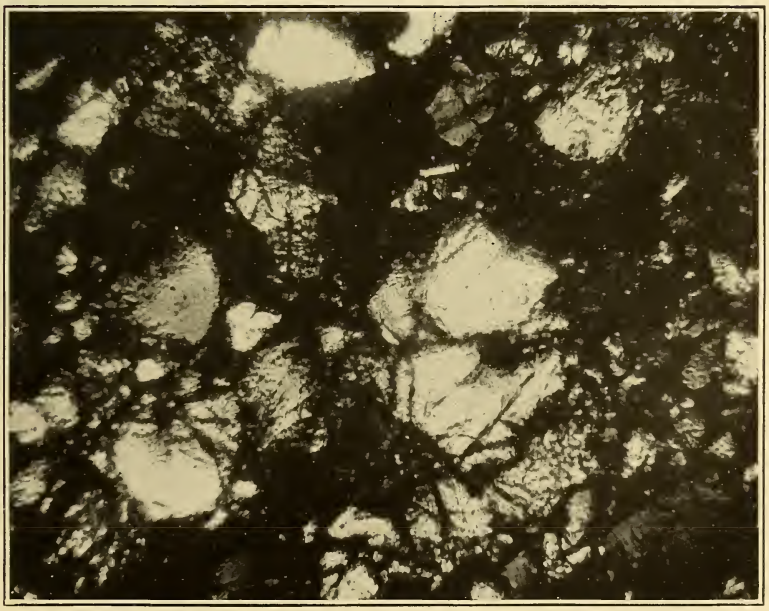

FIG. 28.—*German commercial brick "Stella Werke ☆ ☆ ☆," crossed nicols (magnification 140 diameters), mostly quartz, showing large grains of quartz shot through with cristobalite; practically no tridymite twins*

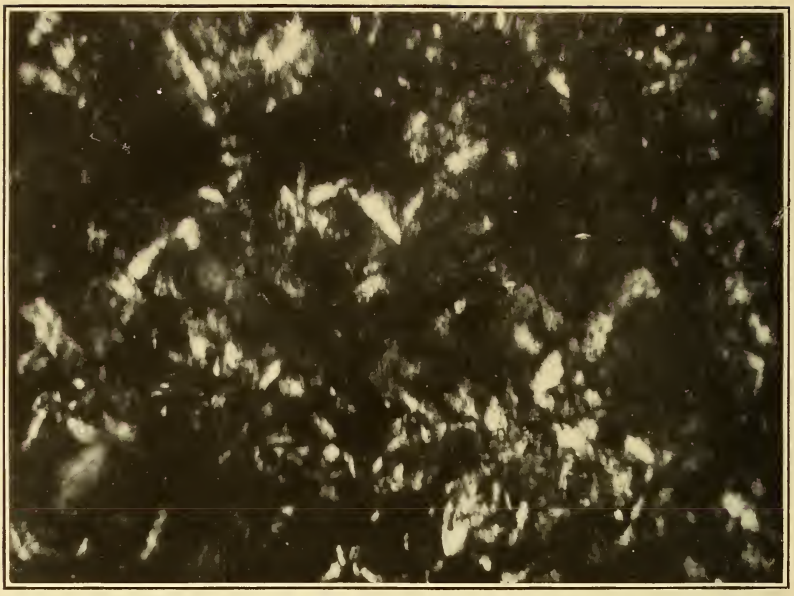

FIG. 29.—*Material of series 1 after third 1500° C. burn. Crossed nicols (magnification 155 diameters), showing many well-developed, wedge-shaped, tridymite twins*

FIG. 30.—*Effect on Medina brick, of flux consisting of 370 parts hydrated lime and 300 parts bauxite, when heated at 1350° C. for one and one-half hours. Shows fluxing away of fine material, which is characteristic of fluxes high in lime*

specific gravity of this material indicate that in manufacture it was held for a considerable period at a comparatively low temperature and was not raised to an extremely high temperature during any stage of the burning.

A sample of material No. 18, Table 16, taken from the heating flue side of the brick has a micro composition as follows: Quartz plus silicates 8 per cent, cristobalite 3 per cent, and tridymite 89 per cent. A sample from the central portion of the brick consisted of quartz plus silicates 20 per cent, cristobalite 13 per cent, and tridymite 67 per cent, while a sample from the coke side consisted of quartz plus silicates 31 per cent, crysobalite 24 per cent, and tridymite 45 per cent. This indicates how the percentage of tridymite increases with the severity of the heat treatment from the coking to the flue side and how at the flue side cristobalite is practically absent. In this case the tridymite is somewhat higher and the quartz plus silicates lower than what McDowell has considered as the condition of equilibrium in bricks made from Medina quartzite. (He gave quartz plus silicates equals 13 per cent, and tridymite equals 87 per cent.)

Figs. 23, 24, and 25 are photomicrographs of regular burned bricks made from standard materials, and Figs. 26 to 29 are similar photos of special material and bricks.

In addition to this work, many of the leading quartzites, including the "loose grained" Medina material and other materials of rather unfavorable character were examined with reference to their value for silica brick purposes. To present the results of this study, it seems advisable first to outline the metamorphosis of silica sand to quartzite. The original quartz of most quartzites evidently consisted of rounded grains, with finer siliceous material and impurities, such as hydrated iron oxide in the interstices. During metamorphosis the fragments recrystallize around the grains and with the same orientation as the grains. Silica deposited from solution as quartz may likewise assume the same orientation as the grains. Silica crystallizing in these ways is pure, except for possible occasional inclusions. Hence in well metamorphosed quartzite, impurities such as hydrated iron oxide, which was mixed with the fine grained material in the first place, will be excluded from the grains in recrystallization and will, under the microscope, appear as sharply defined lines between the hard interlocking grains. Where only a small amount of metamorphosis has taken place such impurities will simply be mixed with the fine silica material and the whole will appear as clouded areas of softer

material between the grains of hard quartz. The tightly interlocking grains of a strongly metamorphosed rock tend to yield a conchoidal fracture and show much more mechanical strength than is exhibited by a slightly metamorphosed material.

It has been found that these strongly metamorphosed quartzites are the ones which produce strong bricks, while the materials that show merely cloudy patches between the quartz grains are those which produce very friable brick or are entirely unsatisfactory.

(*e*) *Effect of Slags.*—Fig. 30 shows the effect produced on a silica brick by a slag consisting of hydrated lime 370 parts and bauxite 300 parts, after having been held at 1350° C for one and one-half hours. In this case it is clearly shown that the fine-grained material was fluxed away first, thus leaving an open framework made up of the coarser particles. This shows up plainly by comparison with the part of the brick not touched by slag.

Fig. 31 shows material which has seen service in the crown of a reverberatory copper refining furnace. In this case the slagging material is largely copper oxide. The surface of the brick which has been exposed shows a porous structure somewhat similar to that of Fig. 30, except that in this case the large particles are also quite porous. The other piece shows how such a slag penetrates the large particles of a silica brick, as well as the finer material.

Figs. 33 and 34 show silica brick treated with slags high in copper oxide according to the method outlined by Nesbitt and Bell.[27]

Fig. 32 shows the ends of two bricks which have seen service in the crown of an open-hearth steel furnace. Although this effect is probably due largely to extreme heat as well as slagging, it is of interest, in that it shows how silica brick act in such furnace crowns when the furnace is carried to too high a temperature (stinging the roof). When so overheated the exposed ends of the brick soften and hang down in long stalactitelike strings.

Comparison of the effects of high lime slags and high iron oxide slags when the slagging tests are conducted according to the above-mentioned method, apparently shows that the high lime slag actively attacks the fine material, slagging it away, while the coarser particles remain comparatively unaffected. The slag high in iron oxide, however, tends to penetrate the large grains as well as the finer material, thus altering the brick as a whole. The slagging materials that come in contact with silica brick in furnace work

[27] Loc. cit.

FIG. 31.—*Brick from crown of copper reverberatory refining furnace, showing how copper oxide slags soak into and alter the entire brick. Piece on left shows depth of penetration; piece on right shows surface exposed to slags*

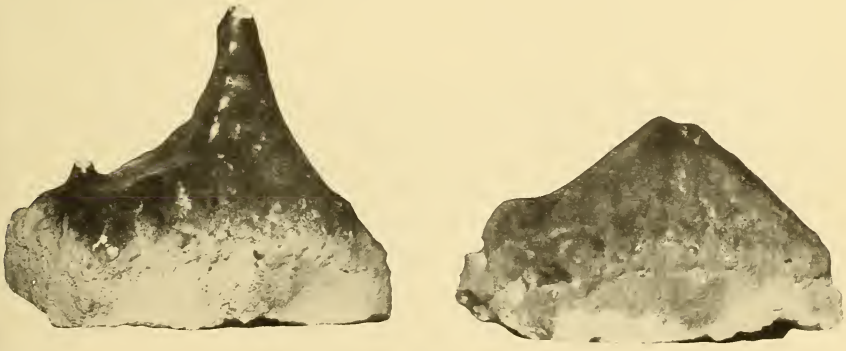

FIG. 32.—*Ends of brick from crown of open-hearth steel furnace, showing melted glassy surface*

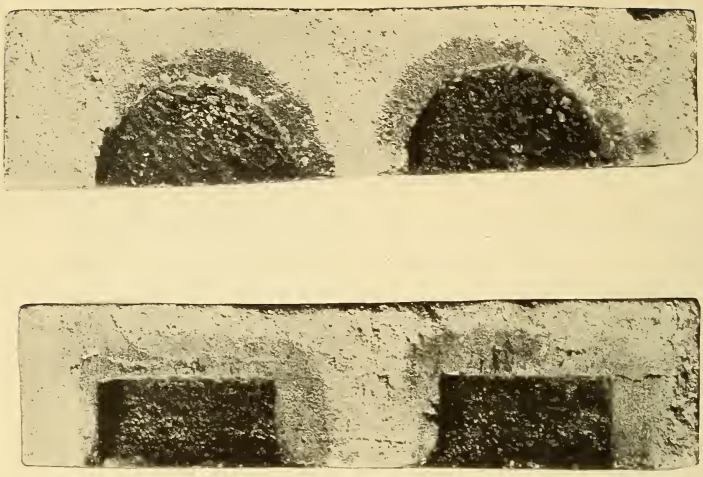

FIG. 33.—*Test made with copper oxide slags by Nesbitte and Bell method, showing nature of surfaces after being acted on by slags. Lower brick was treated with copper reverberatory matting slag, upper brick treated with copper blast furnace slag*

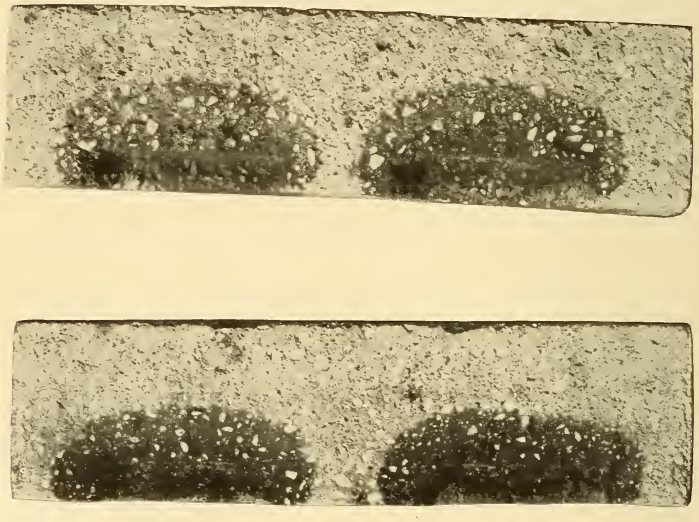

FIG. 34.—*Same pieces and arrangement as shown in Fig. 31, illustrating the amount of penetration of the slags*

are usually of a very basic character, while the brick are extremely acid. Such is the case in steel, copper, and glass furnaces. Such slags will, of course, attack the brick readily. In addition to this many of the slags resulting from such attacking of the brick are very fluid, and have comparatively low melting temperatures. Hence in furnace practice where silica brick come in contact with such basic materials, it is advisable to have the brick as cool as possible by leaving arches, etc., uninsulated.

Slags of this general nature tend first to soak into the fine material of a brick and then gradually into the larger grains. Thus we would expect that the lower the porosity of a brick, the less easily would it be attacked. Also, other things being equal, the greater the proportion of coarse material to fine, the more resistant should be the brick. Likewise quartzites with tightly interlocking grains should be preferable to open-grained material for such purposes.

(*f*) *Screen Analyses.*—Screen analyses and porosities have been determined on 15 unburned silica bricks. These include many of the leading brands and most quartzites that have been used commercially. The data of this work are presented in Table 20.

TABLE 20.—Screen Analyses of Raw Commercial Brick Mixes; Analyses made by the Wet Method

[The porosities of raw and burned bricks of the same brands are also shown]

Screen number mesh	Medina						Baraboo		Homewood formation	
	Number of brick									
	6	2	7	3	5	4	18	21	14	16
	Percentage on screen									
4	1.69	2.44	1.43	1.27	9.97	1.41	3.58	1.72	3.25	11.96
8	19.85	24.18	20.22	14.35	15.63	23.46	27.38	20.98	19.09	
20	11.74	9.67	9.15	10.79	10.01	16.07	15.66	17.86	6.09	6.20
30	3.31	6.36	9.15	9.84	7.76	3.75	5.77	8.84	1.89	4.95
40	10.07	9.67	9.69	15.34	17.23	10.93	4.90	7.97	10.22	29.62
60	18.65	13.97	17.13	16.48	12.17	15.02	12.20	5.73	26.35	16.94
80	1.58	5.70	4.99	3.85	2.97	3.60	2.23	4.24	6.29	2.81
100	3.17	4.26	3.76	3.46	3.27	5.68	3.97	3.32	2.07	5.55
150	4.25	3.33	4.10	2.94	2.32	3.91	3.14	2.60	3.87	1.83
200	3.35	2.45	3.07	2.47	1.51	2.90	2.77	2.76	2.55	1.78
Through 200	9.46	6.63	6.25	5.84	2.88	4.39	8.57	11.27	6.89	8.48
Floated	12.84	11.34	11.06	13.37	14.28	8.90	9.84	12.80	11.38	9.89
Porosity raw brick	26.70	26.15	25.93	27.20	21.22	25.45	23.54	23.20	27.50	28.54
Porosity burned brick	26.35	25.84	28.35	28.52	22.80	27.16	23.91	23.45	30.58	26.55

TABLE 20.—Screen Analyses of Raw Commercial Brick Mixes; Analyses Made by the Wet Method—Continued.

Screen number mesh	Colorado		Missouri	Chert	Eastern Pennsylvania	Average of all varieties (per cent)	Theoretical for minimum porosity[a] (per cent)	Diameter of holes between wires, in inches
	Number of brick—Continued							
	28	27	31	29	12			
	Percentage on screen—Continued							
4	0.23	3.27	0.54	0.37	0.39	2.24	0.40	0.18
8	14.17	16.18	11.09	15.47	21.76	18.85	28.10	.093
20	16.55	22.53	18.15	25.00	22.04	14.52	28.20	.034
30	3.25	12.58	9.90	12.14	6.31	7.06	10.30	.0198
40	4.09	10.06	9.80	4.77	6.45	10.72	4.40	.0150
60	12.05	7.09	11.87	7.55	11.57	13.66	6.10	.0087
80	3.62	1.95	3.43	2.37	3.03	3.51	2.50	.0068
100	3.08	4.10	4.09	3.93	3.81	3.83	2.50	.0055
150	3.92	4.40	3.16	2.78	3.32	3.32	2.25	.0041
200	5.04	4.21	3.03	2.48	2.97	2.89	1.75	.0029
Through 200	21.32	4.09	9.57	11.44	9.83	8.46	13.50	.....
Floated	12.65	9.53	15.37	11.66	8.53	11.56		
Porosity raw brick	26.52	22.93	25.30	24.70	20.32	.....	.....	.....
Porosity burned brick	24.51	24.63	24.70	25.64	23.10	.....	.....	.....

[a] Taylor and Thompson, Concrete—Plain and Reinforced, p. 775. Formula for construction of curve is $d=\frac{P^2D}{10\,000}$, where

D=diameter of largest grain.

d=any given diameter.

P=the per cent of mixture smaller than any given diameter. Largest grain=0.182 inch diameter.

The porosities of burned bricks of the same brands are given for comparison with those of the unburned materials.

In general the mixes that have lowest porosities are those that most nearly follow the theoretical curve for minimum interstitial space, as given by Taylor and Thompson. The data show that as a rule raw Baraboo quartzite mixes have slightly lower porosities and more nearly approach this theoretical curve than do Medina quartzite mixes. Material No. 12 appears to most nearly fulfill this condition, and as might be expected this raw mix has the lowest porosity of all materials tested. On the other hand, sandstones and quartzites, which are but slightly metamorphosed, break mostly to individual sand grains, yielding a great excess of these sizes and a brick of high porosity.

In each case there is an excess of material of sizes that correspond to those of the original sand grains that compose the quartzites. The range of grain size lies chiefly between 30 to 60 mesh, inclusive, for Medina, Baraboo, and Homewood quartzites. In

this respect the chert mix more nearly follows the theoretical curve than do most of the quartzites. This is to be expected since, although the chert shows a granular structure, its grains are very minute. In crushing certain of the quartzites, considerable interstitial bonding material adheres to the original sand grains, while with other quartzites a portion of the original rounded grains break free from the interstitial material, and in still other cases the fracture passes directly through the grains. Microexaminations were made of some of the mixes. Thus in the case of No. 6, which material is from Mount Union, Pa., the silica bond clings to some of the rounded grains, but in most cases the fracture has been through the grains. In No. 21, which is from Ablemans, Wis., some grains break free from the bond and show their original rounded form. This is probably due to the thorough filling of the interstitial spaces, resulting in a uniform bond that is not readily torn asunder. In other cases the fracture is through the grains. There are about equal amounts of whole and fractured grains. In No. 14 there is very little material adhering to the grains. What little amount there is appears to be high in iron. The fracture appears to be frequently through the grains. Nearly all the grains are very angular. In No. 28 the fracture is around the grains in most cases rather than through them. A thin layer of cement high in iron clings to the grains. In No. 31, which is the chalcedony bonded quartzite described elsewhere, the fracture appears to be around the grains and not through them. The grains are all quite rounded, a few grains show attached chalcedony, but more of them are nearly completely covered by a cement high in iron. In No. 12, which is probably Medina quartzite, the fracture is usually through the grains. This may account for the low porosity of the mix.

In considering the porosities of the burned bricks we must keep in mind that all bricks have not received exactly the same heat treatment, hence, in this respect the results are not entirely comparable. Comparing the porosities of raw and burned materials it is seen that the porosity of the finished brick depends quite largely upon the porosity of the raw mix.

Inspection of the screened material revealed the fact that for Medina, Baraboo, Homewood, Missouri, and eastern Pennsylvania materials, the original rounded grains, which broke free from the interstitial material, practically all passed through the 30-mesh screen (0.503 mm diam.), and that but very few passed through the 60-mesh screen (0.221 mm diam.), the great

majority being caught on the 40 (0.382 mm diam.) and 60-mesh screens in varying proportions. In Figs. 35 to 40 are shown samples of materials caught on the 40-mesh screen.

At the present time McDowell is carrying on some studies of the sizes of the built-up, interlocking, grains of several quartzites.

(*g*) *Cold Strength Tests.*—In Table 21 the individual cross breaking tests are shown in pounds per square inch.

TABLE 21.—Cross-Breaking Moduli of Burned Commercial Silica Brick; Bricks Set on Edge with 6 In. Between Supports

Test No.	Medina quartzite							
	No. 6	No. 12	No. 11	No. 7	No. 4	No. 1	No. 5	No. 3
1	1557	205	515	616	279	[a] 312	509	372
2	709	242	448	.....	.....	.....	.....	.....
3	452	243	386	.....	.....	.....	.....	.....
4	1003	190	.....	.....	.....	.....	.....	.....
5	863	270	.....	.....	.....	.....	.....	.....
Average modulus	917	230	450	616	279	312	509	372
Effective modulus	[b] 504	197	385	.....	.....	.....	.....	.....
Specific gravity	2.291	2.480	2.291	2.375	2.393	2.375	2.340	2.424

Test No.	Medina quartzite—Contd.		Baraboo quartzite		Homewood sandstone		Oneida quartzite	West Virginia quartzite
	No. 2	[c]	No. 18	No. 21	No. 14	No. 16	No. 17	No. 38 [d]
1	716	.....	565	467	303	329	1049	664
2	.....	.....	593	.....	472	519	.....	1,143
3	.....	.....	544	.....	.....	.....	.....	782
4	.....	.....	546	.....	.....	.....	.....	645
5	.....	.....	527	.....	.....	.....	.....	809
Average modulus	716	[c] 793	555	467	387	424	1049	809
Effective modulus	.....	.....	530	.....	.....	.....	.....	608
Specific gravity	2.296	.....	2.381	2.390	2.496	2.517	2.274	2.270

Test No.	Montana quartzite	Indiana chert	Southern Illinois crypto-crystalline silica	Average all tests
	No. 25 [e]	No. 29	No. 30	
1	423	446	156	.....
2	.....	.....	182	.....
3	.....	.....	.....	.....
4	.....	.....	.....	.....
5	.....	.....	.....	.....
Average modulus	423	446	169	540
Effective modulus	.....	.....	.....	252
Specific gravity	2.537	2.273	2.363	.....

[a] Sheared.
[b] Determined as shown hereinafter.
[c] For "Star Silica Brick" as given by McDowell (loc. cit.).
[d] Porosity, 28.4 per cent.
[e] Machine pressed.

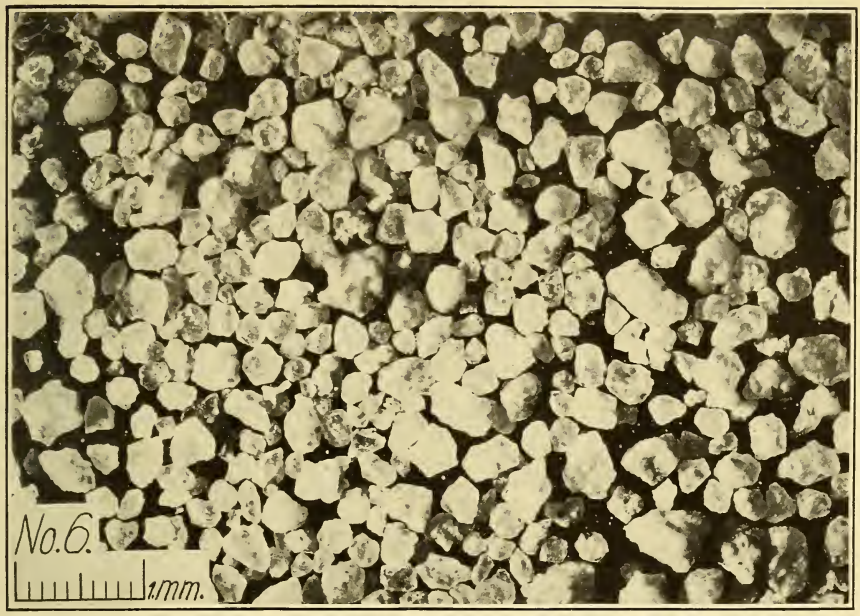

FIG. 35.—*Material on 40-mesh sieve, resulting from wet-screen analysis of raw commercial silica brick mix (magnification 25 diameters). Shows considerable percentage of material that has been broken through the interstitial material between the built-up grains. A slight amount of fine-ground material still adheres to the particles*

FIG. 36.—*Screened and photographed same as material of Fig. 35. Shows large percentage of material broken through the grain, also material in which the breaking was between the original rounded grain and built-up grain. A slight amount of fine-ground material still adheres to the particles*

FIG. 37.—*Screened and photographed same as material of Fig. 35. Original grains of this material appear more angular than is the case with most of the other quartzites examined*

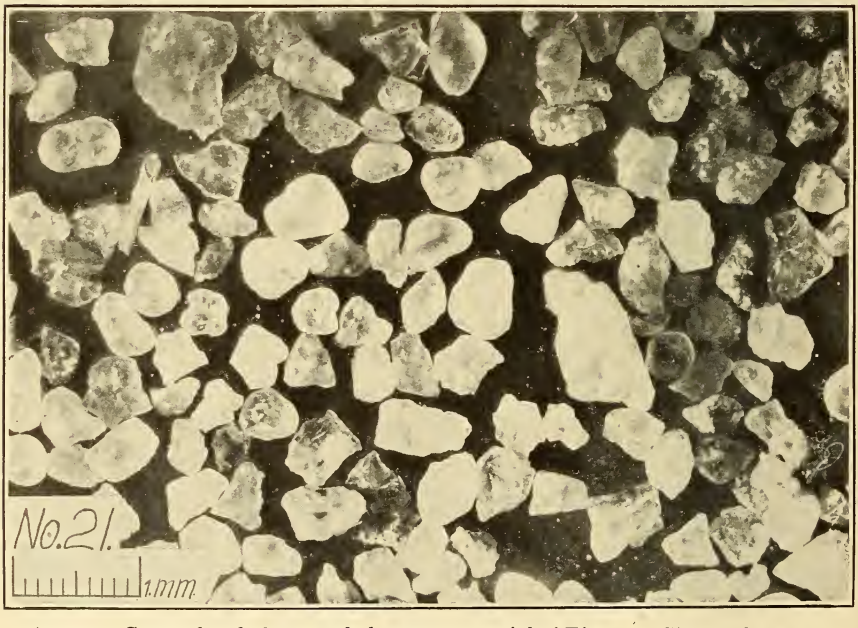

FIG. 38.—*Screened and photographed same as material of Fig. 35. Shows a large percentage of original rounded grains which have broken free from the material of the built-up grains*

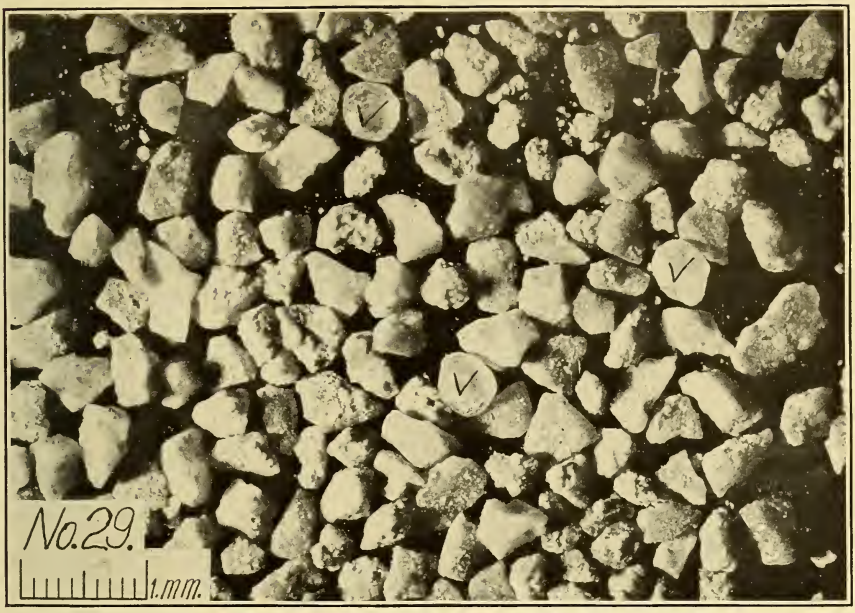

FIG. 39.—*Screened and photographed same as material of Fig. 35. This chert breaks into angular fragments due to absence of definite grain structure. A slight amount of fine-grained material still adheres to the particles. The 3 grains checked (√) are a different material that was accidentally included*

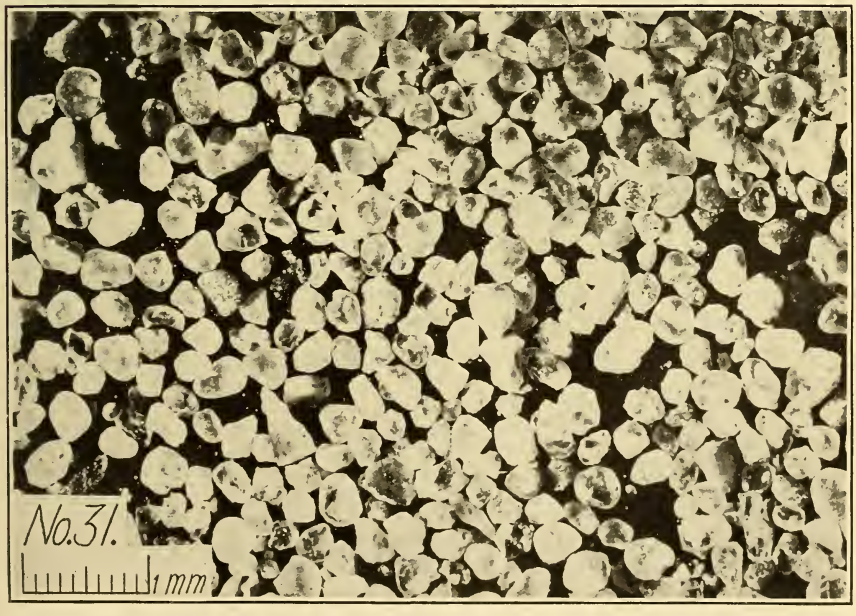

FIG. 40.—*Screened and photographed same as material of Fig. 35. In this material many of the original rounded grains have broken free from the chalcedony bonding material*

It is unfortunate but unavoidable that the specific gravity figures given have not in all cases been based entirely on the brick which have been broken, but in several instances include other brick of the respective varieties.

In considering tests of this nature it must be kept in mind that an average of less than five test pieces can not be relied upon as representative of a material.

Comparison of the cross-breaking moduli and specific gravities in Table 21 indicates that usual commercial burned bricks made from quartzite and in which the quartz has been largely transformed to the crystal forms of lower specific gravity, are stronger than those in which there is still a large percentage of unchanged quartz.

It is interesting to compare the great variation in strength shown by the test pieces of variety No. 6 with the comparative uniformity of those of material No. 18. Thus, although the average modulus for No. 6 is much greater than that for No. 18, its effective modulus is slightly less.

A possible explanation of the nonuniformity of materials No. 6 and 38 may be that the difference in degree of transformation may be considerable between the individual specimens.

The unusually low specific gravities of materials No. 17 and 38 almost puts them in a class by themselves; further, both show high moduli of rupture. This table also permits of some comparison between the bricks made from cherty materials (Nos. 29 and 30). If the test on the single specimen of Montana material (No. 25) is any criterion, we might expect to find that this method of machine pressing has a tendency to increase the cross-breaking strength. However, the abnormality of this specimen may be due entirely to the quartzite used.

The brick tested are average samples from many of the leading varieties used for steel furnace work.

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VI. GENERAL RESULTS

The following statements are based both on the literature cited above and the results of our own work.

1. COMPOSITION[28]

In general silica brick that now give satisfactory service in the United States practically all contain at least 95[29] per cent of SiO_2; many contain at least 1.0 per cent of Al_2O_3, and although few bricks contain much over 1.0 per cent of Fe_2O_3 it has been found that, in certain cases at least, 1.6 per cent did not appreciably depress the softening temperature. Most silica bricks contain 1.60 to 2.0 per cent of CaO. Consideration of the binary system $CaO–SiO_2$ leads us to believe that up to 6 to 8 per cent of CaO the temperature of softening should not be depressed much below 1600° C. Results of our own work on silica brick containing such percentages of lime lead to the same conclusion.

Apparently the limit of total alkalies in first grade silica brick is approximately 0.4 per cent. Increasing the total alkalies above this pointseems to depress the temperature of softening proportionately. The chemical composition of the brick, however, depends primarily upon the composition of the quartzite from which it is made. The desirability of a quartzite depends not only upon a composition that will yield a sufficiently high softening temperature when made into brick, but also upon its physical character.

2. MICRO STUDIES

(*a*) *Raw Material.*—The order of desirability of the different natural forms of silica, according to European authors, is about as follows:

Amorphous silica, including chert and chalcedony, old quartzites derived from the early crystalline rocks, and vein quartz. Quartz schists, sandstones, and sand are usually too friable or impure to have any value. Most of the silica brick used in the United States are derived from the old quartzites, and hence but little is known

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[28] So many physico-chemical factors enter into this problem of effect of impurities that at best it is very complex. For instance, intimacy of mixture, rate of reaction between components, melting temperature and fluidity of resulting products, their subsequent dissolving effect on other grains, the time at any given temperature, and the surrounding temperature, all have their bearing.

The data given under this heading are based primarily on analyses of commercial brands that have given the best service, and upon softening temperatures (as described elsewhere herein), on materials of which the chemical composition was known.

The atmosphere in each softening temperature test was apparently neither strongly oxidizing nor strongly reducing. If other conditions prevailed somewhat different results might be expected. Thus the softening temperature test is a laboratory indicator of quality and not an attempt to reproduce what occurs in commercial furnaces.

[29] This figure is based upon the fact that of the 40 varieties of material tested, the unusual materials noted in Table 16 (Nos. 33, 35, and 36) were practically the only ones that contained less than 95 per cent SiO_2.

in this country concerning the other forms. These quartzites vary in desirability in proportion to their degree of metamorphosis. Thus, other conditions being equal, a highly metamorphosed quartzite in which the interstitial material is in sharply defined bands between the hard interlocking quartz grains is apt to be preferable to one in which the untransformed interstitial material is present in cloudy areas between the original rounded grains of quartz sand. Also medium sized or large grains are probably preferable to fine grained material. This is due to the fact that in crushing they do not readily break to as fine a powder as do smaller grains.

The rates of transformation of the different materials to cristobalite on heating in the neighborhood of 1400° C are probably in the order: Amorhpous silica, chert, chalcedony, and quartz glass. These are transformed at about equal speed,which is rapid as compared with that of quartz. Coarse grained quartzites are the slowest to be transformed of any materials used in the manufacture of silica refractories. Fine material and fibrous quartz which have large surface area are transformed appreciably faster than the large quartz grains. This is probably due to the fact that quartz is transformed to cristobalite progressively from the surface inward and along the cracks.

(*b*) *Burned Material.*—On burning silica brick, which contain the usual 2 per cent of lime in commercial kilns, the silica is transformed first to cristobalite and a small amount of glass composed of silica, lime, and the other basic fluxing materials. When approximately 75 per cent of the silica has been converted, tridymite makes its appearance, and on continued burning practically all of the material is eventually converted to tridymite. However, even in the most advanced material where cristobalite has practically disappeared there is still present approximately 10 per cent of quartz and silica glass. In the average commercial burn we may say that as a rule a few per cent of tridymite has been formed and in the case of a Medina quartzite brick there was found to still be small quantities of cristobalite present, even after approximately 40 short burns to an average temperature of 1450° C. It appears probable that Baraboo quartzite mixes are transformed to cristobalite somewhat more slowly than are Medina quartzite mixes, and also that the chert mixes of the material herein considered are transformed appreciably faster than the Medina mixes. The silicate glass formed in silica brick frequently has a considerably higher index of refraction than any of the

other materials in the brick. This high index indicates high specific gravity. Hence it is possible that in certain cases the specific gravity of a well-burned brick may be raised slightly by the silicate glass present.

3. DENSITY

As the specific gravity of quartz is 2.650; that of cristobalite 2.333; and that of tridymite 2.270, it is apparent that on being transformed from quartz to one of the other forms the density of a brick will decrease. It has been found that except for special materials the specific gravity of the burned bricks always falls within these limits—2.650 to 2.270. A material which exceeded the upper limit was a brick made from a micaceous quartz schist. The high specific gravity in this case was undoubtedly due to the mica present, as mica has a specific gravity of approximately 3. However, material containing sufficient mica to thus raise the specific gravity is outside the realm of best quality silica brick, because of low temperature of softening. The chert brick tested were found to have specific gravities in the neighborhood of the lower limit or slightly below it. The lower limit of specific gravity of chert brick is probably 2.230. The location of the specific gravity of a brick between the limits 2.650 to 2.270 definitely determines the degree to which it has been burned. Of the brick tested the average specific gravity of those made from Medina quartzite is 2.357, and the average specific gravity of those from Baraboo quartizite is 2.399, while that of the commercial brick made from chert is 2.273.

The results of series 2 as presented in Fig. 17 show that at the end of the 1500° C burn the specific gravities of Baraboo, Medina and Alabama quartzites were approximately the same and that there are no very great differences in their rates of transformation, but that between 1300 and 1400° C the Medina quartzite appears to be transformed slightly faster than the other two. The average specific gravity (Table 18) of commercial brick made from Baraboo quartzite is somewhat higher than that of brick made from Medina quartzite. Hence since the principal part of the burning received by the average commercial brick is from 1300 to 1400° C it is probable that at these temperatures Baraboo quartzite actually is transformed slightly slower than Medina. As cited above cristobalite is formed progressively from the surface of the grains and along cracks, and hence most rapidly in varieties having the greatest surface area. In accordance with this we should expect the Medina, which is slightly porous, to be transformed somewhat

faster than the more highly metamorphosed Baraboo quartzite, which is practically nonporous. This effect is probably increased by the lime present.

If we concede such a difference in the rates of transformation, Baraboo brick high in unaltered quartz should be less objectionable in by-product coke oven work, where the average temperature is below 1400° C, than underburned Medina brick would be, since the former are less likely to swell rapidly on being heated.

4. VOLUME AND POROSITY CHANGES

(*a*) *Permanent Changes.*—In the burning of commercial silica brick the volume of the solid material actually increases from 10 to 15 per cent of the original solid volume. At temperatures from 1200 to 1350° C brick mixes containing 2 per cent of lime are transformed (drop in specific gravity) much more rapidly than the quartzites alone. Since transformation goes on rapidly at these and higher temperatures, it is practically impossible to duplicate any given degree of burning except near the minimum specific gravity point, in which case the specific gravities of all varieties approach a common value. Hence, to obtain concordant volume-change results from heating tests, it is advisable to use a heat treatment that will insure minimum specific gravity (one to two hours at 1500° C). However, since the average brick contains approximately 27 per cent of pore space, the exterior volume change will be quite different from the solid-volume expansion. It is practically always considerably greater than the expansion of the solid material. Since this is true the porosity of the burned brick is necessarily greater than that of the raw mix.

Strongly bonded quartizites having tightly interlocking grains yield brick that as a rule are low in porosity and show but small increases in porosity upon being burned, while friable quartzites made up of rounded grains yield brick of high porosity which on being burned show considerable increases in porosity. The average porosity of the commercial bricks tested is 26.34; the average of the bricks made from Medina quartzite is 27.07; and the average of bricks made from Baraboo quartzite is 24.30, while that of brick made from chert is 25.64.

The minimum porosity for a given mix will be obtained in burning by raising the temperature at the usual rate to a temperature between 1250 and 1350° C and holding at this temperature for several days. Thus, transformation of quartz will proceed at a moderate rate and the soaking will permit the fluxes present to exert their

maximum effect in drawing the materials together, thus keeping down the increase in pore space to a minimum. To insure thorough transformation, however, the temperature should finally be slowly raised until a heat treatment approximating cones 18 to 20 is obtained. If, however, a raw brick is heated rapidly to a high temperature transformation of quartz goes on rapidly and undue increase in pore space results.

Tests with Medina brick indicate that well-burned brick suffer far less on being rapidly reheated to 1500° C (six hours) than do medium or underburned brick. To compare directly the volume expansions and changes in pore space of different brick mixes and quartzites on burning, however, the solid volumes and exterior volume expansions should be in terms of the original volumes of the solid-volume materials.

(*b*) *Thermal Changes.*—The chief thermal volume changes in commercial silica brick are those due to the tridymite inversions below 200° C and the Alpha-Beta cristobalite inversion at from 220 to 300° C. The cristobalite change is by far the more important, due both to the greater change per unit of cristobalite present and also to the larger percentage of cristobalite usually present in commercial brick. In underburned brick the presence of untransformed quartz may also cause a slight volume increase at 575° C on heating. It is between atmospheric temperature and 300° C that practically all spalling takes place. The measurement of these volume changes below 300° C offers an interesting problem for future work, as does also the development of a spalling test.

Some work has been contemplated on the measurement of these tridymite and cristobalite expansions in commercial silica brick. It should, if conducted with moderate accuracy, be a fair guide to the amounts of tridymite and cristobalite contained by the samples under test. Incidentally, it would show how the exterior volume expansions at these temperatures vary with the different brands of brick and varieties of quartzite. It might also be used to determine at what temperature between 220 to 300 ° C cristobalite formed in silica brick is most likely to be inverted and cause this volume change. For the cristobalite inversion it would be necessary to hold a fairly constant temperature (approximately 10° C variation) for about three hours, and similarly it is probable that the tridymite changes would require approximately the same time.

A comparatively simple and inexpensive apparatus, which might answer this purpose and which is here suggested, is shown in Fig. 41. With care it is possible to split a silica brick lengthwise through its width and then split one half lengthwise through the thickness of the brick, thus securing a piece approximately 2½ by 1¼ by 9 inches. This reduction in cross section should greatly cut down the time necessary for the brick to reach uniform temperature throughout. Each individual grain of the original quartzite has its own orientation and there are usually several quartz grains in each fragment occurring in silica brick. From this fact and the way silica brick are made, we can depend upon fairly uniform thermal expansion in every direction. The piece

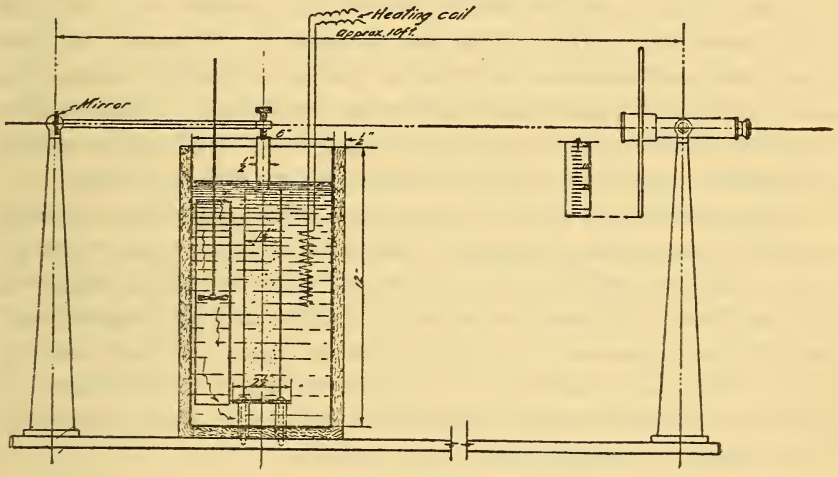

FIG. 41.—*Extensiometer, designed to measure the expansion of silica brick, between atmospheric temperatures and 300° C*

so prepared is placed upon the thin metal or glass plate shown in the sketch, which in turn rests upon quartz glass rods which extend down to the bed plate. A quartz glass rod is placed upon the upper end of the test piece. This rod is of such a length that when the end of the set screw of the lever arm is rested upon it the lever arm may be brought to a horizontal position. When the lever arm is horizontal the mirror should be in a vertical position and the axis of rotation of the lever should lie in its reflecting surface. Thus, by means of a telescope and fixed scale set at a proper distance it should be possible to read changes in length with a fair degree of accuracy. For heating purposes the tank is filled with "Crisco," in which is immersed a nichrome wire heating coil or grid. The "Crisco" is kept in circulation by

means of a small propeller in a tube which is immersed in the liquid. This is necessary to insure a uniform temperature throughout the tank. Any temperature control device which gives a variation of not to exceed 5° C either way should be fairly satisfactory.

Such expansion tests of brick made from the four or five leading varieties of material should be of special interest, as should the time required for standard sized whole bricks to reach maximum expansion.

In load test work rapid heating causes spalling. This spalling frequently results in splitting off corners and edges first, then layers crack off all parts of the surface, leaving a more or less cylindrical portion. This is undoubtedly due to the corners, etc., of the brick becoming heated while the interior is still cool. It was found that certain brick showed no signs of spalling under the same conditions that caused others to spall badly. This makes it appear possible that a rate of heating may exist which would distinguish clearly the spalling tendencies of different brick.

It has been found that, at least with brick made from Medina quartzite, the spalling tendency decreases with increase of tridymite, and that probably coarse mixes spall less than mixes of fine particles. It has also been found that in many cases brick made from Homewood sandstone tend to spall less than Medina brick. This may be due to underburning of the Homewood brick, or to some inherent property of the stone, such as softness of the material between the quartz grains.

5. MECHANICAL STRENGTH

(*a*) *Mechanical Strength, Raw.*—Practically any silica brick material when made up with 2 per cent of lime and thoroughly dried forms a brick that is strong enough to allow of being handled, but a hard material that crushes with just enough fine material to fill the interstices between the coarser angular fragments, makes a much stronger raw brick than does a soft rock or sand that yields a uniform size of rounded grains when crushed. The rules for percentages of the different sizes of material to yield the maximum strength mix in the raw state for any given material are probably very similar to those used in other lines of work that have to do with fineness of grain.

Practically all strength tests to date on silica brick, both raw and burned and in both the cold and hot conditions, show that a tremendous nonuniformity exists in each State. This is true

even in the bricks of a single carload of finished ware. As shown above, this may be quite largely due to the differences in burning which occur in a single kiln of ware. However, the grinding and molding of the bricks may also be partly responsible. It is possible that these latter difficulties might be largely obviated by the following changes in method of manufacture. These changes are not put forth as standard methods which have been thoroughly tried out. They are rather methods that have shown some promise, and that might be used in special cases to overcome the above difficulties. Hence they should be used only after due consideration.

After crushing to 2-inch size the quartzite should be run into a dry pan, containing proper sized holes in the screen plates, and crushed, screened, and the different sizes run into bins from which they could be fed to a mixer similar to that used for mixing concrete, or into a wet pan having raised mullers. Or the material might be crushed directly in a wet pan having raised mullers. The lime should be added at this point. Such a proceedure should make it possible to obtain a practically constant size of particles and the most desirable proportion of coarse and fine materials in the finished mix at all times. When crushed materials are poured from a spout into a bin there is a tendency for the coarse material to segregate from the fine. It is essential to keep this in mind when using bins for storage of ground silica brick materials. A proper adjustment of pan and screens should prevent losses due to an excessive amount of fines, and if found desirable two rocks such as Medina quartzite and Homewood sandstone might be mixed. Or, if desired, a hard and soft material could be mixed together without danger of reducing all the soft material to individual grains, as would be the case with wet pan mixing. The brick should be machine molded, which would insure greater uniformity of texture, shape, and amount of material per brick. Nesbit and Bell [30] have stated that 1500 pounds per square inch is the most desirable pressure for use in the manufacture of machine-pressed silica ware.

As noted in the results of series 7, some of the repressed pieces were friable. This friableness was partly due to fine cracks over the surface of the ware. Such surface cracks appear to be characteristic of machine-pressed silica ware throughout the usual range of pressures (500 to 5000 pounds per square inch). This

[30] Loc. cit.

surface cracking does not appear to be so marked, however, with hard quartzite as with more friable material, and should cause no serious trouble with the average Medina quartzite. In making silica brick by machine, the water content should be slightly less than is the case with hand-molded ware, but care should be exercised not to get the water content too low, as abnormally low water content results in an open-structured brick which lacks strength, both in the raw and burned states. It is probably of little advantage to first mold the bricks by hand and later repress them when partially dry, for although a comparatively large original water content tends to make the particles pack more closely together, the cracks, etc., produced by the hand molding probably more than offset this gain.

(*b*) *Mechanical Strength, Burned.*—In the case of Medina quartzite at least, it has been found that for serviceability a mix of the following screen analysis—residue on 8 mesh 15.3 per cent, 16 mesh 13.4 per cent, 20 mesh 4.6 per cent, 30 mesh 5.4 per cent, 40 mesh 4.8 per cent, through 40 mesh 56.5 per cent—although not always giving the maximum cold-crushing and cross-breaking strength, nevertheless appears to yield satisfactory results. As loose-grained materials make weak raw brick, so also are such brick weak and friable when burned. Brick that are greatly underburned or too rapidly burned are also deficient in strength.

In the cross-breaking test, silica brick practically always fail because of tearing apart of the material below the neutral axis. Hence, this test can be considered a satisfactory measure of the degree to which the materials of a brick are bonded together. This is a rather important matter, as the strength and durability of silica refractories is limited largely by the degree to which the quartz grains are bonded together in the fragments and the fragments are bonded in the brick.

The results of the cross-breaking tests, in conjunction with the natures of the materials broken and the specific gravities, porosities, micro analyses, etc., indicate that with brick made from quartzites, the strength of slightly to mediumly burned brick probably depends largely upon the state of metamorphosis of the raw material, porosity of the raw brick and the lime silica bond formed on burning. Well burned silica brick, however, appear to have their strength somewhat increased by the formation, through

transformation of quartz, of interlocking crystals. Seaver[31] also held that the interlocking crystals of well burned silica brick increased their strength. Or speaking generally we may say that the mechanical strengths of commercial silica bricks, made from quartzites, are inversely proportional to their specific gravities.

In coal gas retorts which are now largely made up segmentally from silica tiles, the question of abrasion is an important one, because of the wear occasioned by continuously charging and emptying. Only the tougher materials appear to be satisfactory. Although but little data are available on the subject, the indications are that the chert brick (No. 29) described above may prove particularly suited for this purpose when burned to low specific gravity. In this work the temperature is seldom extremely high. Hence, certain of the less pure materials, similar to some English silica brick which tend to glaze on the surface or bond more firmly together in use, may also be found valuable for such purposes.

In load test work at furnace temperatures, silica brick seldom contract or show any signs of giving way under a load of 25 pounds per square inch when held at 1500° C for one and one-half hours and have also been found to stand up fairly well with this heat treatment under a load of 40 pounds per square inch. However, in several trials silica brick which were apparently average specimens did not stand up satisfactorily with this heat treatment and a load of 75 pounds per square inch.

In tests in which brick were thoroughly heated to 1350° C; then withdrawn from the furnace; stood on end in a testing machine, and crushed within two minutes from the time of removing from the furnace, after the method of Nesbitt and Bell,[32] approximately 1600 pounds per square inch was found to be the average crushing strength of good quality brick made from Medina quartzite. This would indicate that there is considerable falling off in strength of silica brick between 1350° C and 1500° C. Le Chatelier's figures, above, show this decrease in strength for "star" silica brick. This same quality brick when tested according to the same authors spalling test, shows an average spalling loss of approximately 20 per cent. As brick in the crowns of openhearth steel furnaces are subject to considerable pressure at extremely high temperatures and to wide variations in temperature, the importance of the above tests for brick to be used for such purposes is apparent.

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[31] Loc. cit. [32] Loc. cit.

6. SLAG RESISTANCE

Silica brick are readily attacked by basic materials such as high lime slags, copper oxide, etc. Because of their porosity slags penetrate them readily, and in many cases flux away the fine particles before coarser fragments are much affected. To resist such slags a brick should have as low porosity as possible and contain as great a proportion of large fragments as is compatible with proper strength, etc. Also as quartzites which have tightly interlocking grains are more impervious than those with loose rounded grains, it is to be expected that, other things being equal, the former will resist slagging better than the latter.

7. SPECIAL PROBLEMS

In the course of the present work the following questions arose: First, Can an all-tridymite brick be economically manufactured? Second, What will be the nature of a brick made by the use of 6 to 8 per cent of lime? Third, What will be the effect of only partially transforming the quartz during burning and then using the brick at temperatures not to exceed 1200° C? Fourth, What effect will the sodium chloride present in some coals have upon the silica brick walls of by-product coke ovens?

With reference to the all-tridymite brick, it has been suggested to burn first at a high temperature and later hold for an extended period at a lower temperature. Carrying the brick rapidly to a high temperature will cause an undue increase in porosity, thus appreciably weakening the structure of the brick. As shown above the conversion of cristobalite to tridymite in a silica brick is so slow even at high temperatures that the time would be prohibitive. If the quartzite is strongly calcined, so that it is practically all cristobalite before being made into brick, these brick when burned will have no strength, even when made from quartzites of tightly interlocking structure. Hence even though the resulting brick were largely tridymite it would have no value as such. However, tightly interlocking structured quartzites, when but lightly calcined, made into brick, and burned in the usual way, yield satisfactory brick of low specific gravity. Such a brick should contain a limited amount of tridymite, but is probably far from being all tridymite. Added fluxing ingredients in the amounts tried, although they increase the rate of transformation, appear to cause excessive increase in porosity in about the same proportion and tend to depress the softening temperature.

Hence, they can not be considered satisfactory for this purpose. In summation it may be said that the production of an all-tridymite brick on a commercial basis is very improbable. However, brick made from chert rock should offer some possibilities in this direction.

With respect to the use of 6 to 8 per cent of lime, it is apparent that the speed of transformation of quartz to the forms of lower specific gravity on heating will be greatly increased; hence if abnormal porosity increase is to be avoided, it will be necessary to burn at much lower temperatures than those now in use in commecrial kilns, although a final rather high temperature may be desirable. Such material should not soften much below 1600° C. Hence it might be of value in certain industries where extremely high temperatures do not occur. Such a brick would be favored by the low temperature at which it could be burned. As a rule such brick are not looked upon with favor.

If the quartz in a brick is only partially transformed in manufacture and the brick is then put into use at temperatures up to 1200° C, transformation will continue to take place and the brick gradually expand, except that the effect of soaking in long-continued use at high temperatures may tend to counteract this expansion to some extent by drawing the particles closer together. Material No. 18, Table 16, was apparently an example of this type. It is not reported as having given any trouble.

Certain coals may at times contain small amounts of sodium chloride or other fluxing ingredients, that in part would travel to the silica refractories of by-product ovens during coking. Such ingredients would directly tend to increase the rate of transformation of any quartz present in the brick, thus increasing the rate of swelling slightly.

8. TESTING AND INSPECTING MATERIALS AND WARE

(*a*) *Raw Materials.*—To determine the value of a quartzite for the manufacture of silica brick, it should first be examined microscopically or with a strong hand glass to determine whether the grains are tightly interlocking or not. The grains should be tightly interlocking. If apparently satisfactory it should then be crushed so that the largest particles are one-fourth inch diameter and made up into test pieces containing 2 per cent of lime and given repeated burnings to some definite temperature (between 1300 to 1400° C) in conjunction with other mixes of known worth, simi-

larly made up. The resulting changes in volume of the solid material and of the exterior volume of the pieces, in terms of the original volume of the solid material, as well as the changes in specific gravity, should be plotted as curves. When so plotted the curves will show definitely the rates of transformation of the quartz in the material being tested and the difference between the exterior and solid volume increases will show the increases in pore space of the mixes. In a true quartzite material the specific gravity changes and amount of solid volume increase will ordinarily be approximately the same as those of the standard materials with which it is compared. It is desirable that the increase in pore space be small. This test should of course be confirmed by making up standard-sized brick and burning them in a commercial silica brick kiln.

The softening temperature of the burned mix should not be below that of cone 31. To meet this temperature requirement the original rock must contain approximately 97 per cent of SiO_2 and not much over 0.4 per cent of alkalies.

Amorphous silica, chert, and chalcedony if in solid rock form will be transformed somewhat more rapidly than quartz, but if of sufficient purity should otherwise act much the same as quartz, except that owing to their higher rates of transformation they are apt to crack and fly to pieces if heated too rapidly, thus resulting in weak or ruptured brick.

(*b*) *Finished Ware.*—In considering the inspection of finished ware, it must be borne in mind that the use to which ware is to be put is an important factor in determining their value. Thus, in coke oven work the amount of subsequent expansion of the brick and resistance to abrasion and spalling largely determine the value of a brick. For the crowns of open-hearth steel furnaces the crushing strength of the brick while hot and its resistance to spalling are the all-important factors. For the port bulkheads at the ends of such a furnace and for side walls, resistance to slagging by lime and iron slags is one of the chief requirements. For copper refining furnace crown work the brick must have high hot crushing strength and resistance to slags high in copper oxide.

With reference to the increase in volume on reheating, it is so difficult to obtain check heat treatments that the only satisfactory way of actually comparing the amount of expansion of different varities is to use a heat treatment that will insure minimum specific gravity. In practice, however, this heating is not necessary except as a check. Instead the specific gravity of the brick as it comes

from the manufacturer is taken by means of the wet, dry, and suspended weight method, which clearly denotes the degree of burning which it has received in manufacture, and hence indicates how much it will expand in use.

Since the load that a brick will carry when hot is such an important factor in steel furnace crown work, it appears advisable to have a hot crushing test as well as a load test for this purpose. This might well be done by running a test the same as the load test and at the end of the time increase the load on the brick until it crushes.

In view of the above it is readily seen how difficult it is to give one set of tests that will be fair to all desirable materials and at the same time meet the demands of the various industries. The following is a partial set of specifications. Perforce the values given are approximations, and must not be considered as rigid limits. They should be used rather as general guides to the order of magnitude of the various terms.

Best grade silica refractories:

1. Shall contain not less than 95 per cent SiO_2.

2. When powdered to pass a 60-mesh screen and made into cones similar to Orton pyrometric cones, by the aid of gum tragacanth or similar material they shall not soften and bend over at a temperature lower than that at which Orton cone 31 softens and bends over, when heated (in two hours or less) in a gas-fired hot furnace, in which the atmosphere is approximately neutral.

3. The specific gravity as determined by the wet, dry, and suspended weight method shall not be greater than 2.384.

4. The permanent exterior volume expansion in terms of the original exterior volume when heated to 1500° C as follows: Atmospheric temperature to 800° C, not less than six hours; 800 to 1500° C in six hours; held at 1500° C for one and one-half hours; shall not be more than 6 per cent. (The above heat treatment is just sufficient to lay cone 20 flat.)

5. The transverse strength test conducted on a cold 9-inch brick set on edge on knife-edges 6 inches apart, should, according to the tests shown in Table 21, show an average modulus of approximately 540 and an effective modulus of approximately 284. However, in accordance with the tests on materials Nos. 6 and 18 it appears desirable that the effective modulus of rupture should not be much less than 500. The averages for this figure shall be made up from not less than six bricks. The effective modulus of rupture is obtained by subtracting the average deviation

from the mean modulus of rupture. The modulus of rupture is calculated by means of the following formula:

$$R = \frac{3WL}{2bd^2},$$

where

R = modulus of rupture.
L = distance between supports in inches.
b = breadth of specimen in inches.
d = depth of specimen in inches.
W = load in pounds at which failure occurs.

The mean modulus is merely the arithmetical mean of these figures. Hence where S equals the sum of the squares of the differences between the various observations and the mean modulus, and n equals the number of observations, we have for effective modulus of rupture,

$$\text{Mean } R - \sqrt{\frac{S}{n-1}}.$$

6. The load test shall be conducted in the usual form of load test furnace.[33] The load shall be 25 pounds per square inch. The heating shall be 50° C in 15 minutes from atmospheric temperature to 500° C, 75° C in 15 minutes from 500 to 800° C, 100° C in 15 minutes from 800 to 1200° C, 50° C in 15 minutes from 1200 to 1350° C, and 30° C in 15 minutes from 1350 to 1500° C. The 1500° C temperature shall be maintained for one and one-half hours, at the end of which time the heat shall be shut off and the furnace allowed to cool. When cold the brick shall not have increased in length more than 2 per cent (¼ inch per foot) or decreased in length more than 1 per cent (⅛ inch per foot). (With the above heat treatment approximately Orton cone 19 touches the base after one hour at 1500° C). The refractory materials committee of the American Gas Institute has set 1 per cent permanent expansion as the limit in such tests. This 1 per cent limit is a desirable one if materials can regularly be obtained that will satisfy it.

In case a crushing test of a hot brick is desired, it may be obtained by conducting the test the same as the load test above and at the end of the time loading the brick until it crushes. Another method that has been suggested is to heat the brick to 1350° C at a rate not to exceed 260° C per hour and maintain the final temper-

[33] U. S. Bureau of Standards Technologic Paper No. 7.

ature for two hours. When thus heated the brick is withdrawn from the furnace; stood on end in a testing machine and crushed within two minutes after withdrawing from the furnace.

In addition to the above tests there should be for some purposes a slagging and a spalling test, and for special work an abrasion test. The slagging test suggested in the report of committee C–8 of the American Society for Testing Materials, 1917, and the spalling test suggested by Nesbitt and Bell,[34] are useful, but it appears that further work along these lines might be of value.

The value given above for the specific gravity is the average of the specific gravities of 31 leading varieties of silica brick, and should be used in this sense as a guide, rather than an absolute figure that shall not be exceeded.

VII. SUMMARY

In conclusion it may be said that in general the most satisfactory quartzites for silica refractories are those which contain not less than 97 per cent of SiO_2, nor more than 0.40 per cent of alkalies, and are made up of tightly interlocking grains. The geologic age of quartzites actually used in the manufacture of silica brick in the United States at the present time ranges from Cretaceous to pre-Cambrian. The harder portions of the Medina formation in Blair and Huntingdon Counties, Pa., have proved to be particularly suited for steel furnace work. Approximately 60 per cent of the silica brick manufactured in the United States at the present time are made of this material. The balance comes largely from the Baraboo formation of Wisconsin.

Raw brick made from such quartzites should not show undue increase in pore space on being repeatedly burned in a commercial kiln to approximately 1350° C. Two limiting factors in the use of quartzites are impurity, resulting in low softening temperature, and friableness, which results in weak brick. Amorphous silica, chert, and chalcedony, when in solid rock form and of desired purity may also be satisfactory for the manufacture of silica brick, but as yet have not been thoroughly tried out in the United States. Their rates of transformation on being heated are, however, appreciably greater than that of quartz. Hence they should be heated more slowly to prevent their disruption.

As now manufactured the usual upper limit in the size of fragments present in silica refractories is slightly more than ¼ inch

[34] Loc. cit.

diameter. In the case of brick made from Medina quartzite, those containing coarse fragments tend to spall less than those made of finer material. The slagging effect produced by many basic slags should be kept at a minimum by low porosity, large fragments and by using quartzites having tightly interlocking grains.

It is probable that the nonuniformity of finished brick, due partly to present methods of grinding of mix and molding of bricks may in special cases be somewhat minimized by crushing the quartzite in a dry pan to the proper size, screening, then mixing in proper proportions in a mechanical mixer similar to a concrete mixer, which will not further crush the material, and finally molding by machinery under a pressure of approximately 1500 pounds per square inch. This would also improve shape and finish of the brick.

In the burning of silica brick the volume of the solid material increases from 10 to 15 per cent of its own volume, owing to the transformation of quartz to the lower specific gravity forms of silica, but since the average porosity of such brick is approximately 26 per cent the exterior volume expansion of the brick will be somewhat more than this. The porosity of a well-burned brick is usually greater than that of the raw mix. In silica refractories there is but a small percentage of flux; hence in burning the quartz is transformed directly to cristobalite. However, when approximately 70 per cent of the material has been converted to cristobalite, tridymite makes its appearance and is developed slowly with continued heat treatment, gradually reducing the amount of cristobalite present. A few per cent of quartz plus silicate glass remain, however, even at a very advanced stage of burning. This is probably mostly silicate glass. In the usual silica brick mixes containing 2 per cent of lime the transformation to forms of lower specific gravity is very slow below 1350° C, but above that temperature the rate increases rapidly with increased temperature, and at a temperature of 1500° C a close approach to minimum specific gravity is reached in a couple of hours. With a given heat treatment at temperatures from 1200 to 1350° C the 2 per cent of lime causes the quartz to be transformed much more rapidly than is quartz alone. Greater percentages of lime accelerate transformation still more. The presence of the lime in silica refractories causes their softening temperature to be much more clear cut, because of increased fluidity, than is that of pure quartz.

In burning, to produce a brick of low porosity and low specific gravity, the temperature should be gradually raised to approximately 1250° C and held between this temperature and 1350° C for several days to insure transformation of quartz at a moderate rate, and at the same time obtain the effect of soaking. The temperature should then be gradually raised, so that the maximum of the heat treatment is approximately equivalent to cones 18 to 20. Such treatment should produce a brick containing a considerable percentage of tridymite. However, the production of a brick composed entirely of tridymite appears to be impracticable commercially. Rapid raising of the temperature from 1250° C on tends to produce undue increase in pore space.

Since most commercial silica brick are largely cristobalite, their chief spalling tendency is at the Alpha-Beta cristobalite inversion (220 to 275° C). Also such brick, when heated as in the load test, show a very marked volume expansion at this temperature range, then a gradual but very small additional expansion continues to take place with rise of temperature, until a temperature of approximately 1400° C is reached, at which point expansion due to transformation of any unaltered quartz present in the brick becomes apparent on the expansion curve. This latter expansion will appear as the permanent expansion when the brick is cold. Thus, it is rather difficult to state the coefficient of expansion of silica brick as a single figure.

Since check heat treatments of silica brick are difficult to make, the only satisfactory way of actually comparing the amount of expansion of different varieties is to use a heat treatment which is sufficient to insure approximately minimum specific gravity. The specific gravities of practically all commercial silica brick lie between the limits 2.650 and 2.270. The relative position between these two points of the specific gravity of a brick, as it comes from the manufacturer, clearly denotes the degree of burning which it has received in manufacture, and in conjunction with the porosity indicates approximately how much the brick will subsequently expand in use.

The use of silica refractories is limited largely by the degree to which the quartz grains are bonded together in the fragments and by the degree to which the fragments are bonded in the brick. The cross breaking modulus, which directly measures the degree of bonding, should be of considerable value in determining this quality and the uniformity of silica brick.

The following lines are suggested for future studies on this subject: First, cross breaking strength tests of raw brick made from the five or six principal quartzites; second, hot crushing strengths (1500° C) of burned brick of these varieties; third, expansion of such burned brick during heating to 1500° C; and fourth, studies to determine the changes in tridymite and cristobalite contents of brick, originally high in tridymite (accurately determined by micro analyses) occasioned by long continued heating (several hours) at 1600° C.

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